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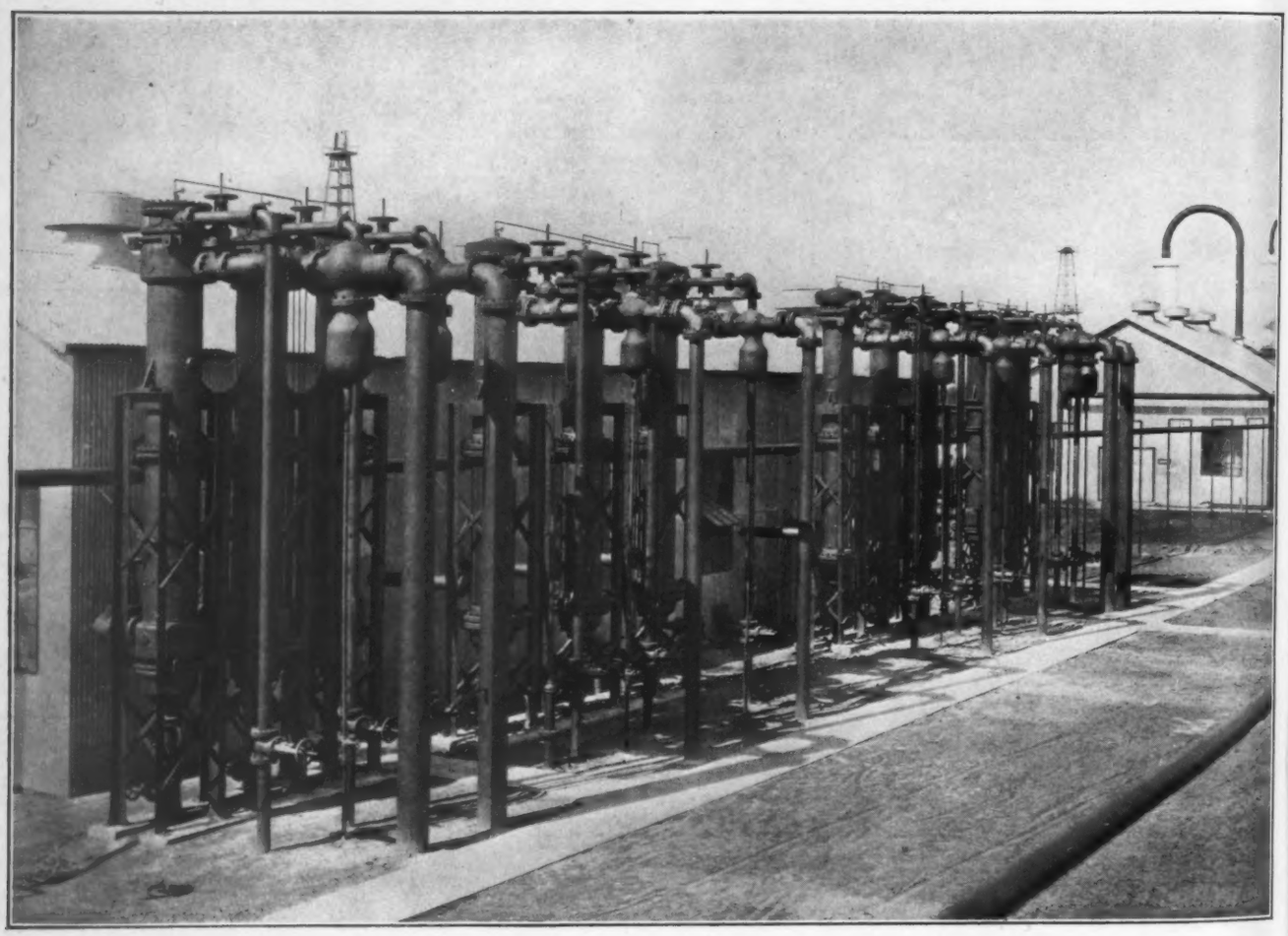

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The REFINER

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Edited Monthly at Tulsa, Oklahoma

GRADY TRIPLETT, *Editor* GEORGE REID, *Associate Editor*

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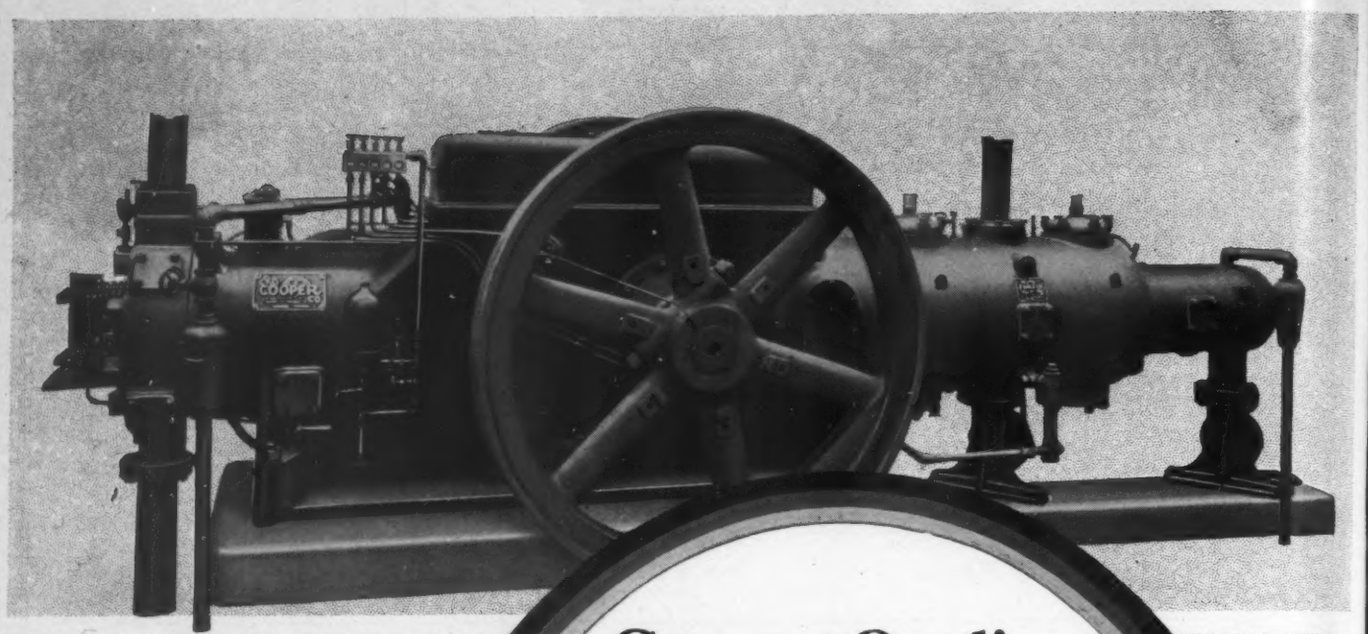

Cooper Quality in a New Small Compressor Unit

The illustration shows the new Cooper Type 16— 50 B. H. P. two-stage compressor unit. The high pressure cylinder is single acting and is an extension of the head of the low pressure cylinder. The whole arrangement is simple, accessible and highly efficient. For boosting small quantities of gas, air or gas lift plants, small gasoline plants or compressing tail gases and weathering gases in refineries this unit is especially adapted.

The gas engine is of the latest four-cycle type with Timken main bearings, drop forged connecting rod with both bearings adjustable, drop forged main shaft, removable power liner, heat resisting valves and self-contained oiling system with filter. The unit is designed heavy enough so that it can be converted to a full Diesel oil engine in the field. Ask any branch office for complete information.

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Sell the Plant Too

THE refinery defies the sample case. An orderly layout of steel, brick and cement, functioning from extreme heat to winter chill, is not to be locked in a portfolio and tucked away under the Pullman berth.

Yet the refining plant is one of the saleable assets of any concern. It is not to be taken along, yet it can be sold and it should be sold. The millions of automobile owners care little about the plant in which it is made. But the thousands of jobbers, who buy gasoline in tank cars have a profitable interest in the plant that is responsible for gasoline.

Each salesman of necessity carries his samples of gasoline and oils, arrayed in neat packages. His specifications and price lists are often good examples of the art of printing. Despite the charm of neatness and color, these are still ordinary, for each salesman has them.

But let a salesman turn from his talk of quality, which is important, to a description of the refinery. He will get an audience that will surprise him. He will have the jobber listening and the office force will drop its work to lend an ear.

The modern refinery is just little enough understood to give it place in the realm of things desirable. Few jobbers ever declined an invitation to visit a refinery. But few of them comparatively ever have the opportunity of visiting a refinery.

This interest in the plant and the fact that few jobbers ever have the privilege of satisfying that interest leave the situation open for the refining concern to sell the plant, to put it in a slang figure of speech.

A few concerns are taking advantage of this condition by furnishing certain representatives with motion pictures of refinery operation. Even this, good as it is, has its limits. A film can not be shown in the jobber's office. A crowd and proper conditions are essential to proper showing.

But each salesman can take the refinery along. With a few ordinary pictures and a store of knowledge of refining methods, a salesman can take any jobber customer on a tour that will be enlightening. Such an effort will get more attention than oil samples and excellent printing. The effort also will assure better attention to the contents of the sample case.

Contrary to the Franklin bit of wisdom as to having cake and eating it, the refining plant can be sold a dozen times each day and it will still remain on the yard, doing its duty from extreme heat to winter chill.

Refining plants should be made to do more than manufacture gasoline. They are a saleable asset. The refining industry is losing an opportunity when it fails to offer its plants.

* * *

Now the Crown

AN EFFORT put forward early in September promises some relief from the Quantity Discount Agreement. There is hope that the Q. D. A. may be abandoned shortly.

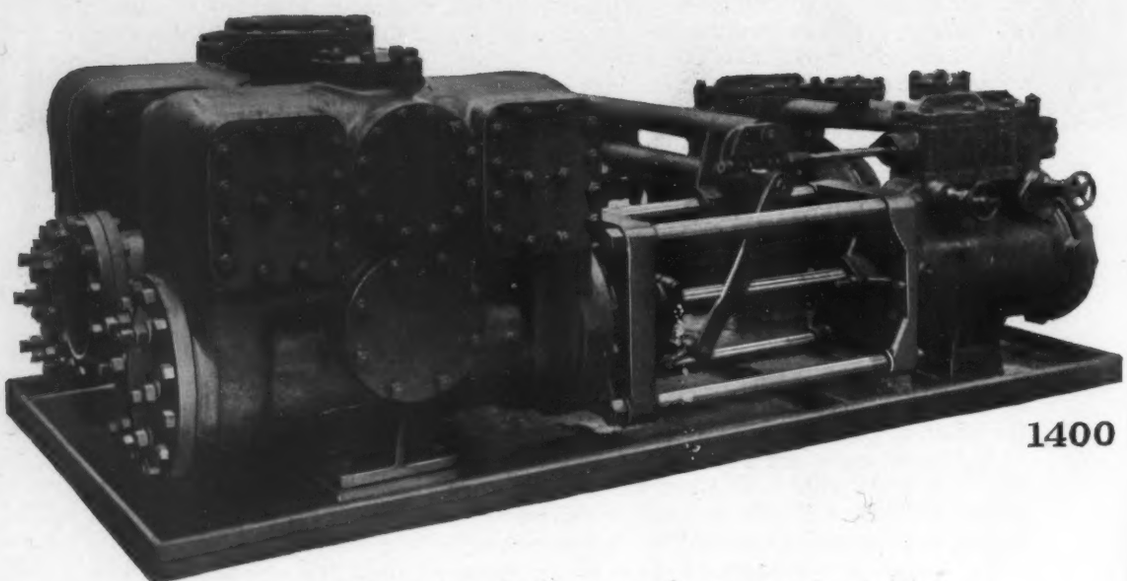
Whatever success comes of this effort, it will be a fitting reward to the era of sensible marketing that independent refiners instituted early this year. Refinery prices have been based on profit for several months.

Now there is a tendency to change bulk plant and service station selling to bring profit into that division of the market.

When profit becomes the foundation of each selling effort in the whole petroleum scheme, refiners will have reached a desirable economic mile post.

Grady Tajbert

Pumping hot tar, asphalt & residuum



12x9x18" Block Valve Pump with base plate drip pan for 300 lbs. working pressure.

While it is true that most any pump will pump oil, it is also true that few pumps will fit refinery service as well as TRANSIT PUMPS, because, like refineries, they are built to handle oil and its products.

For example, consider our Block Valve Type Pumps, illustrated above. We know of several successful refinery superintendents who see to it that all hot tar, asphalt, residuum, and other heavy semi-liquids are handled through TRANSIT Block Valve Pumps. They are convinced that these pumps are built for just such work.

If you have to do with the selection of refining pumps, we will do everything we can to help you get the best pump for any condition.

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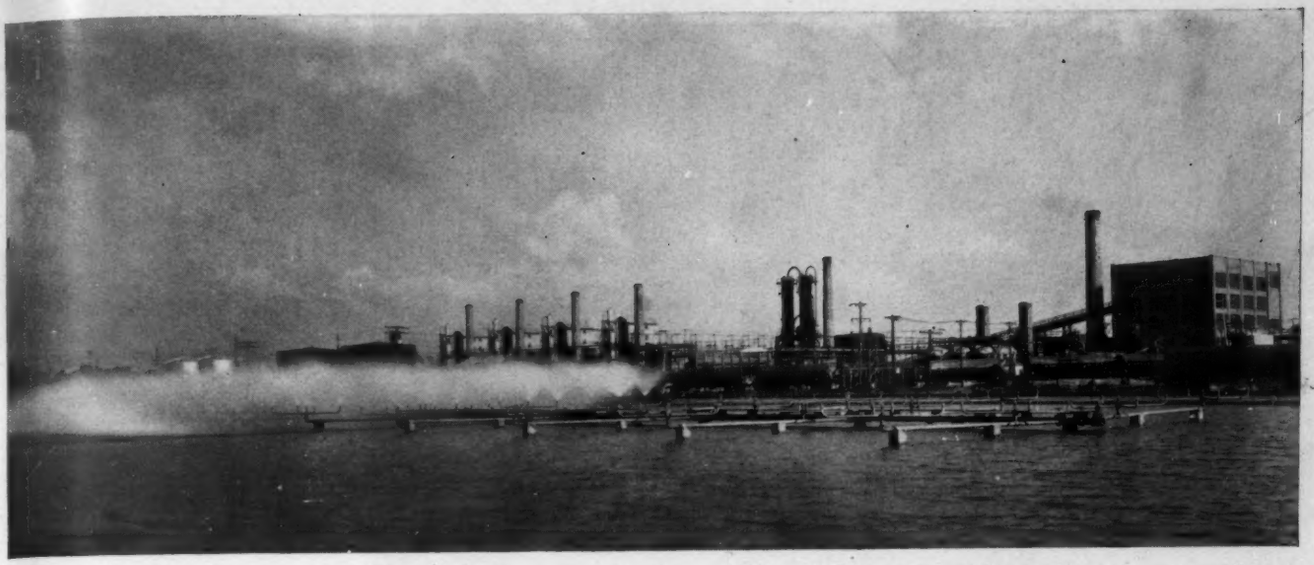


Lincoln Oil Refining Company's Robinson, Illinois, complete plant. The building at the right houses the company's new power plant.

Lincoln Plant Offers Model of Boiler House Control

By GEO. REID
Associate Editor

THE Robinson, Illinois, plant of the Lincoln Oil Refining Company incorporates the latest modern refinery engineering practices in its layout, design and construction. When the present plant was constructed in the winter of 1925-26, much thought was given to the matter of permanency of the installation, as well as economy and efficiency of operation. The units included in this recent program of improvement and expansion consisted of a new power plant, a 6500-barrel daily capacity tubular retort for skimming and preparation of cracking plant charging stock, four Holmes-Manley cracking units, an asphalt plant, and certain additions and repairs to the lubricating oil department and gasoline recovery units.

The pipe still, in which the straight run gasoline is distilled, thus eliminating the use of shell stills for this purpose, was constructed by the M. W. Kellog Company. In this still with one pass through, the crude oil is heated sufficiently to cause the vaporization of all of the gasoline, kerosene, two cuts of gas oil and one wax distillate fraction, all of which are fractionated by bubble type towers working with the stills.

A residium of 15 to 16 degrees A. P. I. is removed from the separator or vaporizer in the usual manner. In addition to control sections at the top of the towers where incoming crude is subject to preheat, the charged oil is further preheated in passing through a large heat exchanger counter-current to the hot residuum. Gasoline vapors liberated in this preheating are flashed into the gasoline-bubble-tower, and the oil is pumped into the tube still for further distillation. The economy of this practice is obvious. The Cross pipe still is shown in one of the accompanying illustrations which shows in some detail the layout and design of this unit.

Five products from the fractionating columns constitute the production of the skimming operation. The heavier cuts, such as the gas oils are blended into charging stock for the four Holmes-Manley cracking units. Gasoline and kerosene derived from the pipe still operation are taken to the other side of the plant yard where they are subjected to such chemical treating and redistillation as is required.

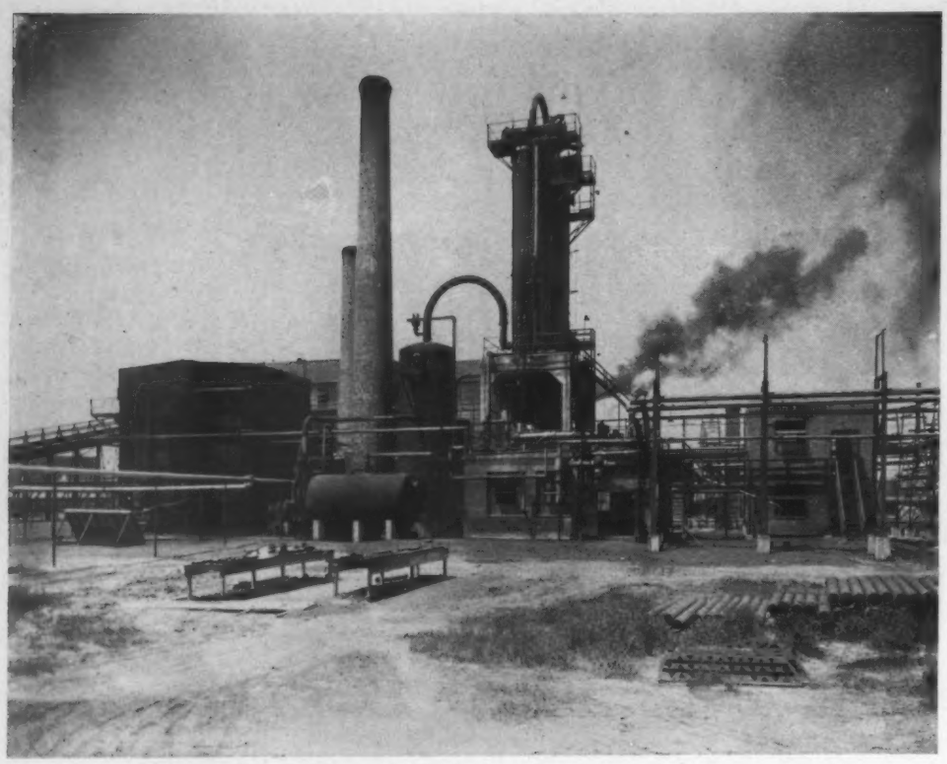
Following the crude flow through the plant in proper sequence it is well to deal with the residuum from the pipe still operation. This material of 15 to 16 gravity is taken direct from the tube still by pump and discharged into a small asphalt plant located near the Cross pipe still. This plant consists of four reducing stills, two of which are run in series, one being used as a batch reducing still where the asphalt is air-blown, and the fourth still in process of cleaning. In this operation the residue is reduced to the proper consistency for the preparation of asphalt to meet a number of specifications. The product yielded overhead is another fraction of wax distillate. This overhead cut is taken to the shell stills.

Rerunning System

There are nine shell stills, four of which operate continuously, and five batch, where all of the plant rerunning is done, such as reducing pressed distillate to viscosity, rerunning certain kerosene fractions, and redistilling pressure distillate. The wax distillate is cracked in these stills, and from there it goes to the wax plant. In the wax plant there is a 25-ton Carbondale refrigerating unit and three Carbondale hydraulic presses, one 48-inch and two 36 inches in size. Leaving the presses the pressed neutrals return to the shell stills for reduction to viscosity.

After reduction the neutrals are conducted to the treating plant where they are given chemical treatment in batch agitators. From the agitators the neutral oils of about No. 7 color are transferred to the filter house. All acid used at the treating plant is reconcentrated to 60 degrees in a Simpson-Mantius acid recovery plant.

Fourteen 10-ton percolating filters using 30-60 mesh fuller's earth handle the treated neutral oils, filtering them to No. 3 color. At the filter house there is a clay burning furnace of conventional type in which the clay gravitates from a hopper at the top of the furnace falling over alternate baffles in direct contcat with the hot gases of combustion. At the bottom of the furnace the roasted earth flows into a 14-inch line through



Pipe still installation at Lincoln Oil Refining Company's plant in which all of the skimming work is done, including two cuts of gas oil and one of wax distillate.

which it is blown into storage bins at the top of the filter house.

Wax from the pressing operation is sweated in the usual manner to 124-126 A.M.P. and is then filtered to color, solidified, and barreled for shipment.

Crude oil processed in the manner described above is delivered to refinery storage tanks by pipe line by the Ohio Oil Company, parent organization of the Lincoln Oil Refining Company. The crude production comes mostly from the county in which the plant it located. This signifies a permanency of crude for plant operation and points to one of the chief of the company reasons for building the new plant along latest engineering lines, using the very best of modern refinery equipment. Other advantages offered at the plant site include such important items as excellent surface and subsurface conditions for building heavy, massive structures and an abundance of good water for refinery and power house consumption. It is also located in a region of comparatively cheap fuel, although much of the firing is done with fuels other than coal, such as coke, heavy residuum, gas and oil. The distillation process is adequately controlled with various types of modern automatic recording control instruments which lend much to the accuracy and ease of operation.

Cracking

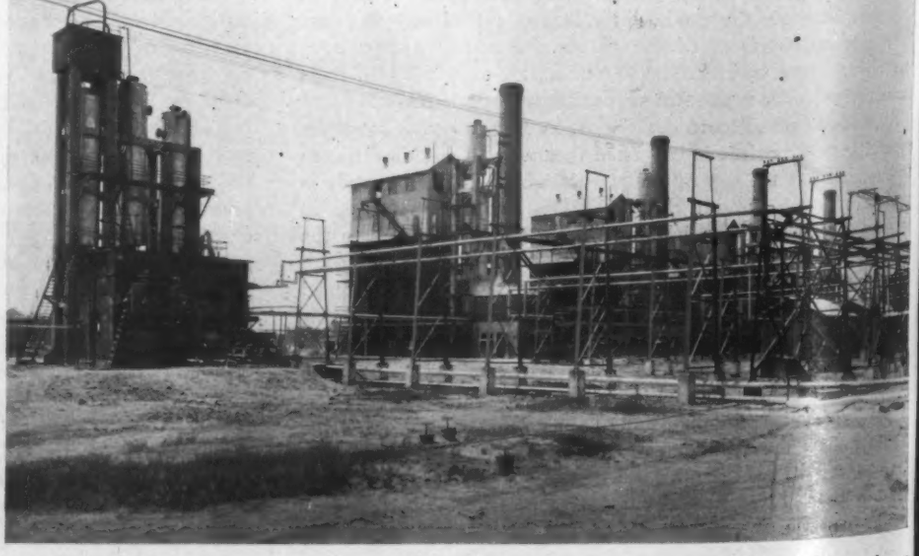
The Holmes-Manley units are charged with a blended charging stock consisting of the gas oils derived from the tube still operation and certain recycle stocks derived from the tar stripper, which will be described later. These cracking units as operated by the Lincoln Oil Refining Company consist of a pipe still or heating element, four vertical stills in series in which the cracking action takes place, a bubble type fractionating column which is surmounted by a dephlegmator, a hot-oil plunger type pump which handles the heavy reflux from the bubble tower, condenser, and pressure release tank where the gasoline and gases produced are accumulated.

In the pipe still the charging stock is passed through a preheater section first where it is heated slowly by the coolest gases of combustion en route to the stack. From this section or tube bank, the oil passes into the heating section, passing progressively into hotter zones until it reaches a temperature of around 805 to 810 F. as it leaves the coils.

Leaving the tube still the oil and vapors pass into the first of the vertical stills and flow through the series of four connected stills. The oil level in the still is controlled by means of the transfer lines from one still to another. Above the oil flow lines are vapor transfer lines through which the vapors pass to finally leave the fourth still and pass into the bubble type fractionating column, through the dephlegmator, then to condensers and accumulator or pressure release tanks.

The four vertical stills are set in a furnace with the top and bottom ends protruding from the heated section. This furnace is mildly heated by gas burners set in the walls, which effects a maintenance of the temperature of the heated oil and gives the time of exposure to cracking conditions of heat and pressure which is one of the important factors in cracking.

The stills are housed in a corrugated iron building, which gives the unit an appearance different from many other cracking plants now so extensively employed by the industry. Each of the stills is equipped with an electric motor driven stirring



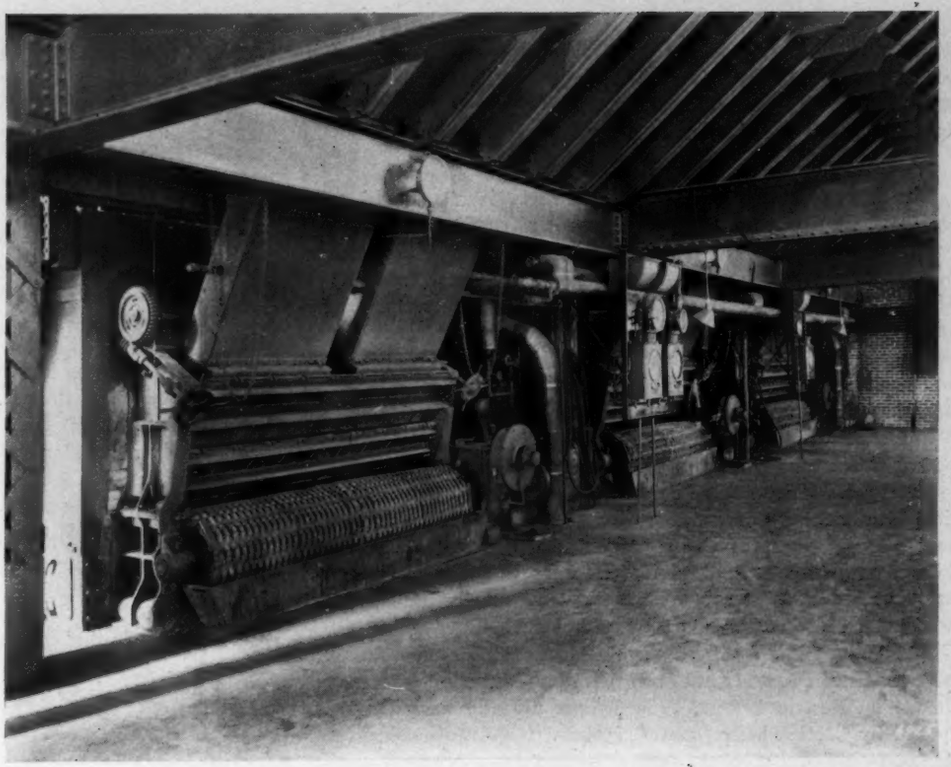
Battery of four Holmes-Manley cracking units with tar stripper or flashing system shown at the left.

and scraping device, driven by spur gears and pinions and mounted at the top of the stills. The scraper keeps the walls of the stills clean so as to retard the deposit of coke. Most of the coke is deposited in the fourth or last still in the series.

Tar Stripper

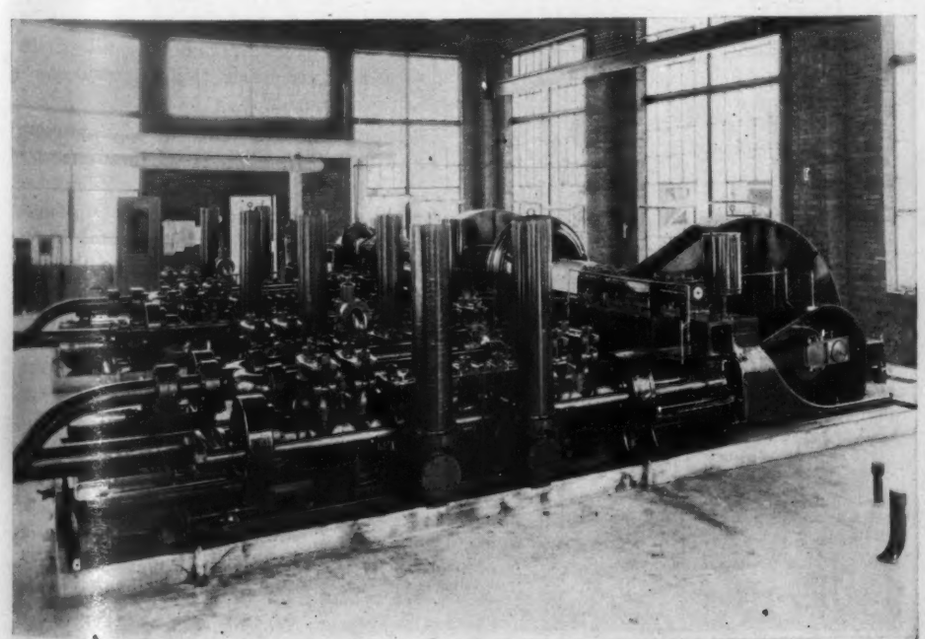
Each of the four vertical stills in series is connected at the bottom to a common header line by means of nipples and valve, and each still is "dragged" or blown out at periodic intervals, through this header line to a flash column which is termed the "tar stripper." The system is not unlike the usual flashing system extensively employed on other cracking units in mechanical design and it effects similar results. When the valve is opened at the bottom of the individual still the tar or residuum is blown out under the plant operating pressure, into the header line and conducted to the flash column at a temperature of approximately 800 F. In the flashing chamber much of the tar vaporizes and the vapors are conducted into the bottom of a fractionating column, leaving the flash column at the top. Leaving this column these vapors pass upward through the second column which is surmounted at the top with a dephlegmator. From this point the vapors are led to condensers and storage. This product is a heavy naphtha. The bottom of this second column yields a distillate which is called light recycle stock. Going back to the first column or flashing chamber, a bottom stream is conducted from it to another fractionating tower. This material is flashed with steam, and an overhead stream of heavy recycle stock is cooled and stored. From the bottom of this last tower is taken the heavy tar or residuum.

The Two recycle stocks are blended together and when needed are transferred to cracking unit charging stock storage tanks or working tanks and are blended with the raw charging stock derived from the Cross pipe still, and in this manner are utilized for further cracking. The tar or residue derived from the tar stripper is utilized as fuel at the plant. A Schutte-Koerting heavy fuel burning system for use in firing the Holmes-Manley units has been installed. This consists of two insulated tanks with loop line leading from this storage to the burners on the line. The heavy fuel is excellent for this purpose. It is kept hot and kept circulating. It is fed to the burners mechanically instead of being atomized by steam.



Interior of boiler control showing installation of mechanical chain grate stokers feeding small coal to the furnaces.

One pump is used to charge all four of the cracking units. Two Goulds piston type pumps are installed, one being an auxiliary unit. One pump handles the charging stock to the units at the rate of 372 barrels per hour. Another pump is driven by an electric motor, and the other, or auxiliary, is driven by a steam turbine. Control of the flow of oil from this single pump to each of the cracking units is handled by the operative in the control or receiving house, by means of remote control valves.



Charging pumps for the battery of cracking units. One pump charges all units.

The gasoline accumulating from the operation of the units is transferred from the control house to the other side of the plant where it is given a caustic wash to remove hydrogen sulphide and it is then stabilized before being doctor treated. In this process the lighter gases are separated, compressed and the available gasoline recovered. The permanent gases are used for fuel.

In this connection it may be well to mention that the plant is equipped with an efficient gasoline recovery plant which handles vapors from the Cross pipe still, the rerun

stills, and many of the light oil storage tanks, excepting those which are equipped with Wiggins floating roofs. The plant consists of two C. & G. Cooper gas engine driven compressors, size 17½ by 20, and an absorber. This recovery plant has an average daily production of around 3000 gallons of gasoline.

Power House

The power plant at the Lincoln refinery is a splendid example of modern power house engineering from both the viewpoint of mechanical construction, and economy and efficiency of operation, and enhances the common boiler house to a thing of real beauty. The single large building of concrete, brick and steel, houses both the steam generating and electric generating equipment. The house is well lighted and ventilated, and equipped with latest type of modern equipment all of which is automatically controlled with an enviable precision.

The steam generating plant consists of three Vogt 500-horsepower water tube boilers, which are equipped with Coxe chain grate stokers steam engine driven and installed by the Combustion Engineering Corporation. These stokers feed the small coal into the front of the furnace and convey it to the rear, where, as ash, it is dumped into hoppers where it is continuously cooled by a stream of water. Periodically the hoppers are dumped into trucks or gondolas and hauled away.

The boilers are equipped with water cooled walls, radiant heat superheaters, and with both forced and induced draft fans, driven by electric motors. The boilers are automatically controlled by Carrick boiler control system. This method includes the actuation of a mercury column by the steam pressure which acts directly on the dampers and the draft. The performance of the boilers is constantly recorded by the Bailey steam-flow, air-flow system, which indicates every detail of the condition of the boilers in operation. They are also equipped with Bailey draft gauges. All control instruments are centralized at the front of the boilers within sight of the man in charge of their operation.

Illinois coal, termed No. 5 Carbon, grading from three-eighths-inch lumps to dust, and inch and a half screenings are utilized as fuel. Some coke is mixed with the coal at present. An inclined tipple runs from the ground to the coal bunker at the top of the boiler house. An electric motor driven winch just above the bunker pulls the loaded cars to the top of the boiler house by means of a steel cable. The cars are then dumped into the storage bins, from where the coal is fed direct to the boilers by gravity.

Two of the boilers are operating all of the time and the third is auxiliary, or stand-by. The company contemplates the early installation of a fourth boiler, which it plans to equip for burning powdered coal, gas, or oil. The boilers usually operate at about 250 per cent. above their rating.

Generating Plant

Steam from all of the boilers passes into a large header at 250 pounds pressure and 150 F. superheat. This header line conducts the steam into the electricity generating plant, where it passes through one of two Westinghouse two-stage steam turbines operating at 3600 r.p.m. The turbines are direct connected to Westinghouse 1250 K.V.A. 440 volt generators. The header delivering this steam to the turbine is manifolded in such a manner that as much as is needed of this high pressure steam can be by-passed for plant use. Further, steam can be bled off of the turbine after the first stage at 125 pounds pressure for the process of other plant uses and is returned at one or two pounds pressure to the second or low side of the turbine. Thus the steam at 250 pounds passes through the high pressure side of the turbine and is taken to the plant at 125 pounds, and exhaust steam from the plant is returned to the turbines through a 20-inch header line, well insulated, which extends through the plant yard to various departments. It returns at two pounds pressure, passes through the low side of the turbine and into a condenser.

But one turbine is used at a time. Both turbines are set on bases supported by concrete pillars and separate from the concrete floor of the boiler house by a space about one-half inch wide. This prevents the vibration from damaging the cement floors, etc.

The turbines are full condensing, with the condensers installed on the floor below. The condenser system is held under 27 to 29 inches mercury vacuum. All condensed water from the turbines, as well as the water from traps in steam lines about the plant, is returned to the boiler water supply tank as distilled water. On account of the large quantity of condensed water returned to the boilers through this system, but very little make-up water is needed for the plant operation. The feed water goes to the boilers at about 200 F. The full condensing feature of the turbines presents an important advantage where distillation conditions are more or less constantly changing, for it makes possible satisfactory adjustment of the steam load.

All Steam Metered

Steam used in all of the various departments of the refinery is metered to the different parts of the plant through Bailey steam meters. In this manner the steam requirements of all of the various processes are accurately determined.

The utilization of high pressure steam which is reduced to 125 pounds in the turbines while generating electricity for the operation of the plant machinery, and the return of exhaust steam to the generating plant for further utilization in propelling the turbines effects the generation of this power at a lowered cost and effects a marked reduction in the volume of steam required for plant operation by its replacement by electric motors.

The executives of the Lincoln Refining Company in charge of the refinery and the other branches of the company are J. K. Kerr, president; H. B. Carpenter, vice president; W. B. Filson, secretary; F. A. Billstone, treasurer, and R. E. Luton, general superintendent.

Installation of steam turbine driven generators producing current for power and lighting for the entire plant.

Results of Study of Adsorptive Power of Clay

By R. O. MEADOR

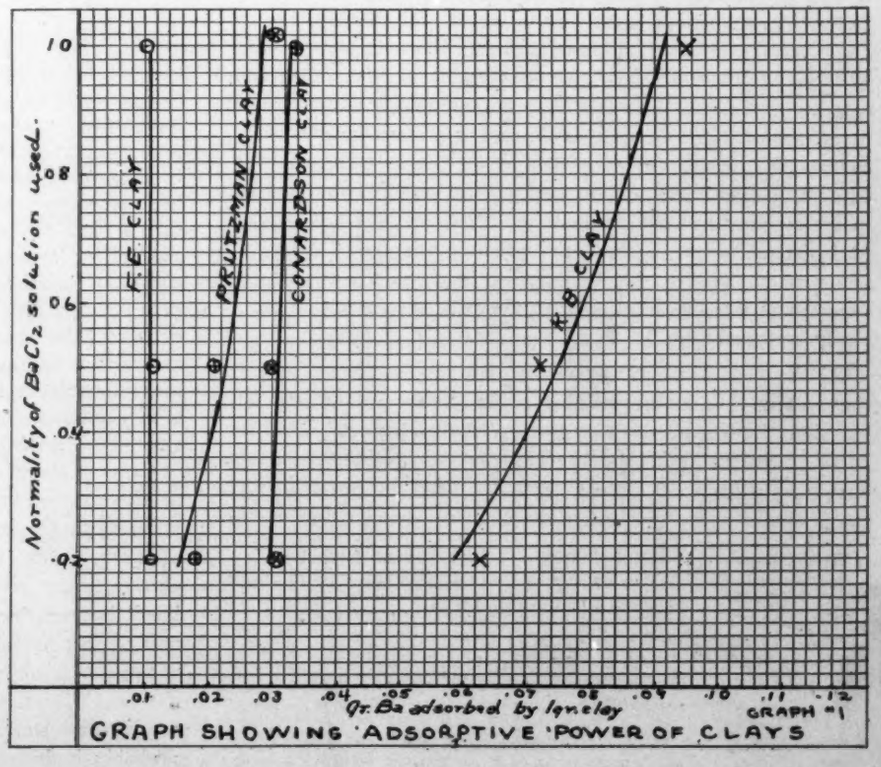
THE theory that a solid body when introduced into a solution removes from the latter a part of the solution is generally understood as adsorption. Bancroft[1] states that the decolorizing action of charcoal has been known since 1791. It is therefore evident that adsorption, as such, has been known for a long time.

Probably the best authenticated hypothesis concerning adsorption is advanced by Freundlich[2], who considers that in the phenomenon just defined as adsorption the free layer of the solution possesses a concentration differing from the body of the solution; that where adsorption is possible, the change in concentration always takes place with a resulting great decrease of the surface tension; and that, if the latter is decreased in the solvent by an increase in the concentration of the dissolved substance, the latter will try to concentrate in the surface layer. Freundlich explains "positive adsorption" as being due to the fact that this concentration in the surface layer is limited only by diffusion back into the solution, and that if the surface tension of the solvent is increased by the solute, then "negative adsorption," or a decrease of concentration of solute in the surface layer, takes place. By the surface layer is meant every surface that divides the solution from some other substance, either solid, liquid or gaseous. Hence, if a solid body is introduced into a solution, a new surface is created, and if the surface tension of the solution at the surface of contact with the introduced substance is so influenced by the dissolved substances that it becomes smaller with the increase in concentration of the latter, then an increase in concentration of the dissolved substances will take place at the surface of contact. If the solution is removed from the solid body, the layer with the high concentration of solute remains attached to the solid body. In other words, the solid body has "adsorbed" a portion of the solute, and the solution that remains is now less concentrated than it was originally. This, according to Freundlich, is the primary behavior of the adsorption process. His explanation is physical and not at all chemical. It remained for Gurwitsch and others to show that very important secondary processes doubtless follow.

No Chemical Combination

Graefe[3], Guiselin and Handicourt[4] and Cross[5] are among those who regard the decolorization of petroleum products by hydrosilicates primarily as a physical and not a chemical process. Lob[6], on the other hand, is an ardent supporter of the chemical theory of adsorption. He considers adsorption as a chemical reaction due to the supposed presence of some substance in the silicates that combines with the coloring matter of the oil and thereby renders it insoluble.

Gurwitsch[7] is of the opinion not only that adsorption is primarily a physical process, but also that in certain cases it is accompanied secondarily by purely chemical processes, such as polymerization of unsaturated hydrocarbons. That no chemical combination takes place between the decolorizing agent and the adsorbed substance was shown by Gurwitsch by treating a benzine-soluble pertoleum product with a decolorizing powder, extracting the powder with benzine and then examining the extracted oil. It was found that the color of the oil recovered from the extract was still lighter than that of the original and that the quantity of the extract was considerably smaller than the quantity of the oil taken out by the powder. Although a part of the oil substance was so firmly retained by the powder that benzine would not extract it, it could be extracted with other solvents, such as ether and benzene, until free from the constituents of the adsorbent, thereby proving that there was no chemical reaction or combination. Furthermore, Gurwitsch found no indication of soap formation when naphthenic and stearic acids were adsorbed by hydrosilicates, an additional proof that in the adsorption of petroleum products, primarily, there is no indication of chemical reaction. However, in aqueous solutions, adsorption can be accompanied by chemical reaction between the adsorbent and the product adsorbed. Gurwitsch further states that the adsorption of paraffin, aromatic and lubricating-oil hydrocarbons is a reversible process, for, after treating these hydrocarbons with a hydrosilicate, benzine will extract these oils quantitatively and in an unchanged condition. In the case of asphaltic and bituminous bodies, however,



[1]Bancroft—"Applied Colloid Chemistry."—McGraw-Hill (1921).

[2]Freundlich—"Kapillar Chemie."

[3]Graefe—Petroleum, 3, (1907), 292.

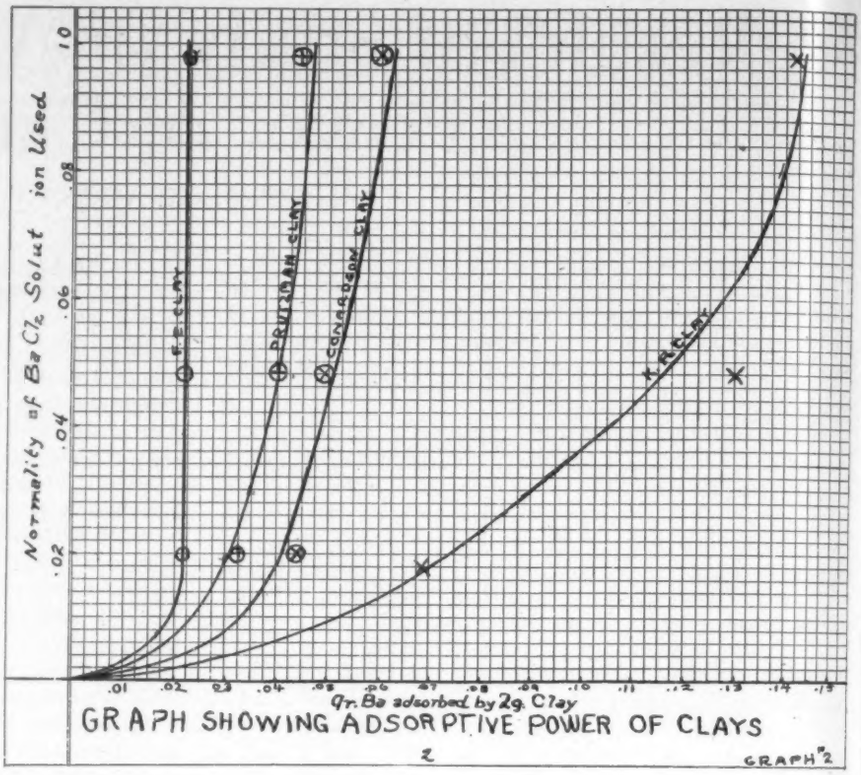
[4]Guiselin and Handicourt—Mat. grasses, 5, 2815, 2845, 2882.

[5]Cross—Bull, No. 16, "Handbook on Petroleum, Asphalt, and Natural Gas." Kansas City Testing Laboratory (1922).

[6]Lob—Chem. Rev. Fet-Harz-Ind. 15, 80.

[7]Gurwitsch — "Wissenschaftliche Grundlagen der Erdolbearbeitung" 1912).

their adsorption is irreversible, and it is therefore to be assumed that they undergo certain secondary changes during adsorption. That a polymerizing action takes place was shown by Gurwitsch by the fact that he was able to change amylene to diamylene by the action of hydrosilicates. That unsaturated compounds of petroleum also undergo similar polymerization in conjunction with adsorption, was proved by the fact that oil treated with Floridin, after being combined with all the recovered extracts removed from the powder by benzene, ether and solvents, had a darker color than the original oil. That various intermediate conditions are possible between the very loose force of ordinary freely-reversible adsorption and the tight, absolutely-irreversible, "truly-chemical" combination was shown by the fact that when Floridin was used in sufficiently large excess in the adsorption of naphthenic acids, the benzine used in the extraction of naphthenic-acid-laden Floridin flowed off practically neutral; while ether or benzene, on the other hand, extracted the adsorbed naphthenic acids quantatively.



Both Chemical and Physical

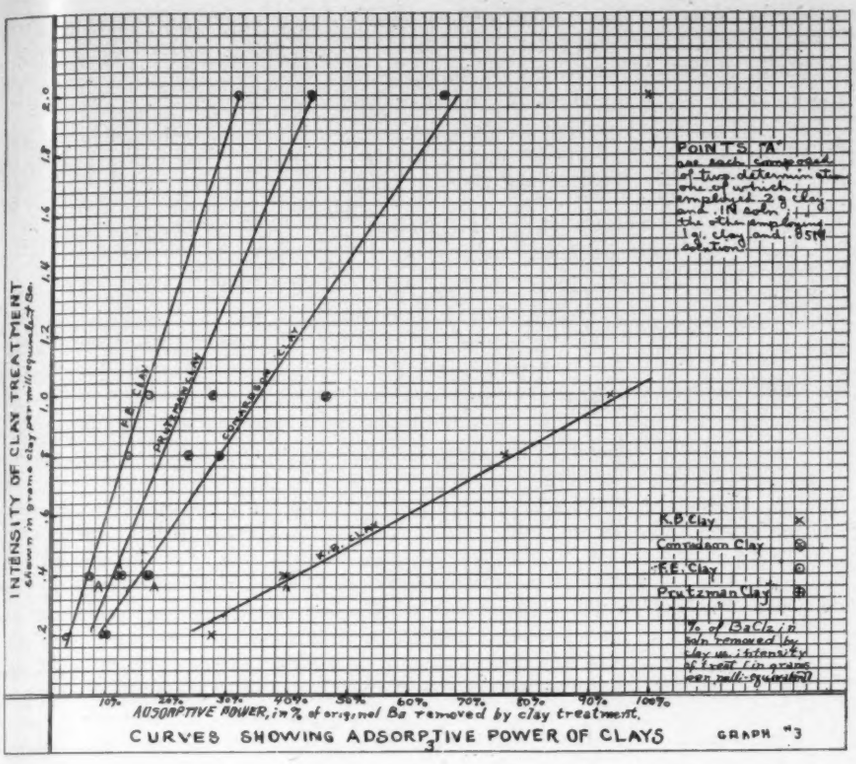
Bell[8] is of the opinion that decolorization of petroleum products is both a chemical and a physical reaction, the chemical reaction being indicated by a material increase in temperature between the incoming and the outgoing oil at the start of the run, Morrel and Egloff[9] likewise believe that fuller's earth exerts a chemical as well as a physical effect in refining, particularly in the purification of cracked hydrocarbon oils. Gilpin and Schneeberger[10] regard fuller's earth as a dialyzing septum and petroleum as an emulsoid, dialysis allowing the free passage of paraffins, and causing the coagulation and adsorption of the bitumens, such action also causing entrainment of the sulphur and nitrogen compounds, as well as such olefines as are present. Day[11] recommends that a quantitative study of the pure hydrocarbons of different series be made in order to show their relative adsorptive capacity; for the difference between the capacities of the various constituents to concentrate on clay surface is so great that it is possible to remove practically all of certain constituents from solution (or colloidal suspension) in the other oils of the mixture. Furthermore, the oils poorest in hydrogen seem to show the greatest adsorptive capacity; while among oils of the same series (such as the oils of the paraffin series), those of greater viscosity are somewhat more easily removed.

Of the finely porous substances that are in general use at the present time in the refining of petroleum products by adsorption, the various sorts of hydrosilicates, or so-called fullers earth, constitute by far the largest group. It has been definitely shown by many investigators[12] that the bleaching power of any hydrosilicate is independent of its chemical composition. However, of general interest are the results of J. Porter[13], showing the following chemical constituents of various sorts of Floridin:

	Per Cent
Silica	44.0 to 72.0
Alumina	5.0 to 33.0
Iron oxide	1.2 to 15.0
Calcium oxide	0.3 to 7.4
Magnesium oxide	0.4 to 4.3
Sodium & potassium oxides	0.4 to 8.3
Water	4.3 to 25.0
Sulphur trioxide	Small Amount
Phosphorous pentoxide, etc	Small Amount

The great adsorptive power of the hydrosilicates depends primarily on their internal structure. Gurwitsch[14] has found by comparative examination of finely-ground Floridin in ordinary and in polarized light that even the smallest particles of the finely-ground earth are not massive, but possess a very fine microstructure; and that it can be considered as practically certain that the inhomogeneous Floridin particles are honeycombed by innumerable microscopic canals that bring about an enormous increase in the adsorptive surface. Gurwitsch further believes that the power of adsorption belongs to a large extent to the amorphous part of the hydrosilicate, basing this assumption on the fact that purely crystalline calcium carbonate, although its individual parts are much smaller than those of Floridin, nevertheless, at least in regard to petroleum, has very little adsorptive power; likewise, barium sulphate and other very finely crystalline powders are of little value insofar as the decolorization by adsorption of petroleum products is concerned. Of interest in the above connection is the opinion of Porter[15] that the clarifying action of fuller's earth is due chiefly to the presence of colloidal silica. Maynard and Mallory[16] believe that the bleaching power of fuller's earth, like many other colloid peculiarities, is due to a combination of adsorption and mechanical filtration, accompanied by some chemical disintegration due to selective adsorption. These writers also state that the active constituents of the earth are probably hydrous silica and hydrous aluminum silicates; and

[8]Bell—"American Petroleum Refining." Van Nostrand (1923).
[9]Morell and Egloff—The Refiner & Natural Gasoline Manufacturer. 2, 9 (1923).
[10]Gilpin and Schneeberger—Am. Chem. Jour. 50, 59-100 (1913).
[11]Day—"Handbook of the Petroleum Industry." (1922).
[12]Among these, Phalen-Chem. Met. Eng. 21, 469 (1919).
[13]J. Porter—Chem. Revue, No. 10 and 11 (1908).
[14]Gurwitsch—"Wissenschaftliche Crundlagen der Erdolbearbeigung" (1912).
[15]Porter—Bull, 315, U. S. Geol. Survey, 1907, 268.
[16]Maynard & Mallory—Chem. & Met. Eng., Vol. 26, (1922), p. 1074.


INTENSITY OF CLAY TREATMENT
ADSORPTIVE POWER, in % of original Ba removed by clay treatment.
POINTS "A" are each composed of two determinations one of which employed 2 g. clay and 1N acid and the other employed 1 g. clay and .85N solution
K.B Clay
Conradson Clay
F.E. Clay
Prutzman Clay
% of Ba Cl2 in soln removed by clay vs. intensity of treat (in grams per milli-equivalent)
CURVES SHOWING ADSORPTIVE POWER OF CLAYS GRAPH #3

that the porous nature of the earth is due to the fact that it is built up of grains approaching colloidal size.

Opinions Differ

From the above review of the bibliography on the subject, it it at once evident that opinion differ among investigators as to the reaction that takes place when constituents in a hydrocarbon oil that give color to it are removed therefrom by an adsorbent material. The general concensus of opinion, however, seems to be that the reaction is primarily a physical one, followed by certain secondary chemical reactions that take place at least in the case of certain particular types of hydrocarbons.

Whether or not the adsorptive power of different hydrosilicates (clay) toward a typical base, such as barium, varies in direct proportion with the decolorizing values of the clay was the basis of some investigational work by the author.

Four clays were quantitatively examined for barium adsorption under mutually identical conditions. The types of clays tested are shown below:

Clay No. 1—*200-mesh, acid-treated San Diego Bentonite (Montmorillonite.) Raw clay was ground treated with 50 per cent by weight of sulphuric acid, water-washed, dried and ground to the fineness indicated. (Marked in Figures, "K. B. Clay.")*

Clay No. 2.—*120-mesh, untreated Colorado fullers earth of low decolorizing value. (Marked in Figures "Conardson Clay").*

Clay No. 3.—*Same clay as No.* 1, *but, after acid treatment and water-washing, clay was not subsequently dried; instead, it was applied to the oil to be decolorized in the form of a clay-pulp containing approximately* 30 *per cent solids. (Marked in Figures "Prutzman Clay.")*

Clay No. 4.—*200-mesh Florida fullers earth. (Marked in Figures, "F. E. Clay.")*

Apparatus and Method

As the investigation was conducted on a quantitative basis, only C. P. chemicals, thoroughly-mixed samples of clay and distilled water were employed in the work. All volumetric apparatus was carefully re-calibrated. One and two gram samples of each clay were added to 50 cc. portions of 0.10, 0.02 and 0.05 N (normal) barium chloride and the twenty-four mixtures resulting analyzed, according to the following procedure:

The Babcock bottle containing the mixture to be analyzed was shaken for 15 minutes at 70° F. It was then laid aside at room temperature (70° F.) until the supernatant liquid had become clear. Twenty-five cc. of this clear liquid were then pipetted out, and the barium that remained unadsorbed in this twenty-five cc. portion thereafter precipitated as barium sulfate, which was filtered off, ignited and weighed. From the weight of barium sulphate obtained, the weight and percentage of the original barium adsorbed by the clay were calculated.

Acidity and alkalinity determinations of each clay were made by titrating one gram samples, while constantly agitating the clay at 100 cc. of distilled water, against either standard caustic soda or standard sulphuric acid solutions as was needed. Phenolthalein was used as an indicator during the titration.

Moisture determinations were made on each clay by heating a one-gram sample for 12 hours at 260° F. and noting the loss in weight. Sample No. 3, which was applied to the oil in the form of a clay-pulp when making the decolorizing tests, and which originally contained 70.2 per cent of water, was dried to a moisture content of 1.5 per cent for use in making the barium-adsorption tests.

Table 1 gives data as to the results of some of the miscellaneous, preliminary tests that were made.

TABLE I

	Clay No. 1	No. 2	No. 3	No. 4
*Decolorizing value, considering fullers earth as 1.0 in efficiency. Results are calculated on a net-dry basis.	3.0	0.32	4.8	1.0
Loss on ignition (called moisture in calculating above) per cent	19.3	10.85	8.0	15.8
Moisture on clay as used (loss after heating for 12 hrs. at 260° F.) Per cent	11.8	2.1	1.5	3.4
Acidity, in cc. of tenth normal potassium hydroxide required to neutralize one gram of clay	12.9	0.0	4.7	0.34
Alkalinity, in cc. of tenth normal sulfuric acid required to neutralize one gram of clay	0.0	0.3	0.0	0.0

*Samples 1 and 2 were heated in a closed flask with outlet for escape of steam, to a maximum temperature of 350° F.; samples 3 and 4, in a similar manner, to a maximum temperature of 400° F.

Table 2 gives full data as to the result of this investigational work.

TABLE 2

	Clay No. 1	No. 2	No. 3	No. 4
Grams of original Ba. adsorbed by 1. g. clay from 50 cc. 0.1 N soln.	0.0950	0.032	0.033	0.0106
Per cent of original Ba. adsorbed by 1 g. clay from 50 cc. 0.1 N soln.	27.7	9.3	9.5	3.0

Intensity of treat, ignoring concentration, in gms, clay milliequivalent Ba.	0.2	0.2	0.2	0.2
Grams. of original Ba. adsorbed by 1 g. clay from 50 cc. of 0.05 N soln.	0.072	0.030	0.021	0.0115
Per cent of original Ba. adsorbed by 1 g. clay from 50 cc. of 0.05 N soln. 42.0		17.6	12.0	6.8
Intensity of treat, ignoring concentration, in gms. clay per milliequivalent Ba.	0.4	0.4	0.4	0.4
Grams of original Ba. adsorbed by 1 g. clay from 50 cc. of 0.02 N soln.	0.063	0.032	0.019	0.0116
Per cent of original Ba. adsorbed by 1 g. clay from 50 cc. of 0.02 N soln. 93.3		46.1	27.4	16.9
Intensity of treat, in grams clay per milliequivalent Ba. ·	1.0	1.0	1.0	1.0
Grams of original Ba. adsorbed by 2 g. clay from 50 cc. 0.1 N soln.	0.142	0.060	0.044	0.023
Per cent of original Ba. adsorbed by 2 g. clay from 50 cc. of 0.1 N soln. 41.4		17.5	12.7	6.7
Intensity of treat, in grams clay per milliequivalent Ba.	0.4	0.4	0.4	0.4
Grams of original Ba. adsorbed by 2 g. clay from 50 cc. 0.05 N soln.	0.130	0.049	0.040	0.023
Per cent of original Ba. adsorbed by 2 g. clay from 50 cc. of 0.05 N soln. 76.0		28.3	23.6	13.5
Intensity of treat, in grams. clay per milliequivaent Ba.	0.8	0.8	0.8	0.8
soln. 99.0		65.2	43.3	31.1
Intensity of treat, ignoring concentration, in grams clay per milliequivalent Ba.	2.0	2.0	2.0	2.0

Data given in Table 2 are shown also in graph form in Figures 1, 2 and 3.

Conclusions

1. No direct ratio between decolorizing values and barium adsorption value of clays was noted.

Grams of original Ba. adsorbed by 2 g. from 50 cc. 0.02 N soln.	0.068	0.044	0.030	0.022
Per cent of original Ba. adsorbed by 2 g. clay from 50 cc. 0.02 N				

2. The possibility that such a relationship holds was not lessened, because the conditions of the decolorizing tests were not identical with those of the barium-adsorption tests. Instead, decolorizing tests were made in a manner that endeavored to duplicate plant practice in the contact filtration of oils.

3. A definite intensity of treat, for example one gram of clap per centiequivalent barium, gives a definite percentage adsorption regardless of the barium chloride concentration (see paragraph 3, points A, A, A, A.)

This insignificance of concentration precludes the view that the disappearance of barium is due to an equilibrium metathesis (in which the equilibrim point would be a function of barium concentration). The data tend to indicate that the disappearance of barium is due to either (1) a unimolecular reaction in which saturation of a definite adsorbing power of the clay is the governing factor, or (2) a completed metathesis.

4. It is believed that if comparative figures obtained under mutually identical conditions were obtained, a definite conclusion as to the nature of the relationship between decolorization and adsorption could be reached.

Natural Gasoline Association Adopts Vapor Pressure Testing Method

THE Natural Gasoline Association of America, through its secretary, A. V. Bourque, announces the adoption of the Reid Vapor Pressure Testing Method as tentative standard by the Natural Gasoline Association of America, effective September 1, 1928, and submits the following statement in connection therewith:

INTRODUCTORY

The Natural Gasoline Association of America has considered for some time the adoption of a satisfactory vapor pressure testing method for the products of the natural gasoline industry. Everyone is aware of the fact that the present I. C. C. method is far from being satisfactory, when one considers its use for specification purposes. The Technical Committee of the Association and several of its members have been investigating for several months the various vapor pressure testing methods which have been proposed by different people. After a thorough investigation and a careful consideration of all proposed methods, the Technical Committee concluded that the Reid and the Beistle-Prather were the two outstanding methods. Accordingly, the Technical Committee recommended the consideration of these two methods to the Board of Directors of the Natural Gasoline Association of America. The report of the committee in this connection is included later in this article. The Board of Directors decided to adopt the Reid method, noting the following advantages of the Reid method over the Beistle-Prather method:

1. It is less liable to breakage on account of the absence of gauge glasses.
2. It is less liable of leakage for the following reasons:
   (a) Pressure during test is always above atmospheric regardless of the volatility of the sample being tested. Leaks under vacuum are difficult to detect.
   (b) There are fewer connections.
3. Calculations involved for vapor pressure determinations with the Reid method are more simple than those encountered with the use of the Beistle-Prather method and apparatus.
4. Determinations are made in a shorter length of time.
5. The apparatus costs less because it is more easily and more readily constructed and repaired.
6. Vapor pressure determinations can be made on material with a vapor pressure less than one atmosphere without the use of a vacuum gauge or a compound gauge.
7. Avoids particular manipulative difficulties attendant to the Beistle-Prather in tests of gasoline having a vapor pressure less than one atmosphere.
8. It is more easily manipulated and, therefore, better adapted for everyday use at loading racks.

The Board of Directors having adopted the Reid vapor pres-

sure testing method the Technical Committee tentatively standardized the apparatus and method of procedure. The tentative standard is also included in this article.

The Association wishes to stress the point that the primary purpose of standardizing a vapor pressure method at this time is to provide another and rational means of establishing the true value of natural gasoline. It should be borne in mind that Reid vapor pressure values can not be substituted for the present I. C. C. vapor tension va'ues. The association is not prepared at this time to definitely recommend transportation vapor pressure limits on the new basis but feels that sufficient information is available for establishing satisfactory limits.

Report of Technical Committee

Engler Distillation Characteristics—Many parties had suggested that the Engler distillation could be used to indicate the vapor pressure of natural gasoline. For this reason the first matter considered was this possibility. The following conclusions were reached:

1. It would be inadvisable to base vapor pressure requirements on Engler distillation characteristics as expressed by the standard grades of natural gasoline. This is because our standard grades are susceptible of change. Perhaps the most far-reaching change in grades in the past was changing the temperature of the receiving cylinder bath from 60 to 32° F.

2. The Engler initial boiling point is a fairly reliable indication of the vapor pressure in the case of gasolines of similar bases. However, the use of the Engler initial boiling point is too greatly complicated by room temperatures, variations in composition of gasoline, personal factors, etc. to be a reliable guide to vapor pressures.

Members of the committee had conducted tests which comprised weathering a given gasoline and thus establishing fairly good relationships between Engler initial boiling point and vapor pressures by the various methods. It has even been possible to establish definite relationships between the Engler initial boiling point and the existing I. C. C. vapor tension values in many such limited experiments. In practice, however, serious discrepancies are invariably encountered. Attention was called to the facts that room temperature corrections would always have to be used; barometric and altitude corrections would be extremely complicated; and specifications concerning the matter of determining Engler initial boiling points would have to be much more carefully written than they are. The committee feels that even with all these complications provided for, the Engler initial boiling point would still be an unsatisfactory indication of vapor pressure. The committee is aware of the fact that very satisfactory relations between vapor pressure and initial boiling points have been observed when the initial boiling point was obtained by placing the thermometer in the boiling liquid. This relation holds very well for the ordinary grades of motor fuel but is not very satisfactory for the more volatile grades of natural gasoline.

3. The Engler distillation loss is a fairly reliable indication of the vapor pressure, particularly in the case of gasolines of similar bases. The Engler distillation loss is not subject to personal error to a great extent, room temperature does not materially affect it, and the method of determining it is very carefully specified. Barometric pressure affects it pronouncedly. The biggest factor against the use of Engler distillation loss is the fact that radically different natural gasolines have the same Engler distillation loss.

Attention is called to Figure 24, p. 127, of the December, 1927, A. P. I. Proceedings where the following ranges in "true" vapor pressure are noted for gasolines having given Engler distillation losses.

Engler Distillation Loss (%)	"True" Vapor Pressure Range Lbs./Sq. In. Gauge @ 90° F.
5	4.5 to 11.5
10	9.0 to 15.5
15	11.5 to 18.5
20	14.0 to 21.5
25	17.0 to 24.5

These discrepancies are believed to be due entirely to the innate differences in the gasoline tested. Because they exist the committee concludes Engler distillation loss can not be used as a vapor pressure specification.

4. The greatest possibility in regard to the use of the Engler distillation as an index of vapor pressure of natural gasoline lies in a correlation of intermediate boiling points, distillation loss and perhaps gravity. This would require considerable further work and should be given serious consideration. The committee feels that this use of the Engler distillation can eventually be made as acceptable as any other means of determining vapor pressure. This fact should be kept in mind in any recommendations in regard to vapor pressure methods.

Since it is evident to the committee that this correlation will require considerable time and thousands of experiments, it is believed an improved vapor pressure method should be adopted at this time. Attention is called to the fact that the adoption of an improved vapor pressure method will permit the desired correlation in the ordinary progress of business.

Vapor Pressure Methods—(a) The expression "true" vapor pressure has been used in this report. The committee wishes to make it a matter of record that its conception of the "true" vapor pressure of natural gasoline is a reasonable approximation of the vapor pressure of the natural gasoline itself so determined as to eliminate the effects of vapor pressure exerted by foreign air, dissolved air, non-hydrocarbon gases and as small a percentage of the fixed hydrocarbon gases as is consistent with commercial application. One definition that has been used for "true" vapor pressure of natural gasoline is the vapor pressure of the gasoline calculated from its composition analysis assuming that the "propane and lighter" in the natural gasoline is all propane.

(b) To state that the present I. C. C. vapor tension method yields results which frequently cannot be duplicated is only to reiterate what all previous committees have stated. All parties interested are in accord in this respect.

(c) As far as indicating the "true" vapor pressure of the gasoline, the present I. C. C. method is of no value.

Reference is made to Figures 3 and 4, P. 108 and 109, of the December, 1927, A. P. I. Proceedings, where vapor tension values by the present I. C. C. method are plotted against "true" vapor pressures. The extremely erratic nature of these values when compared with "true" vapor pressures is self-evident. The following table shows the ranges in comparative vapor tensions noted in these plots:

Temperature (°F.)	Present I. C. C. Vapor Tension (Lbs./Sq. In. Gauge)	Range in "True" Vapor Tension (Lbs./Sq. In. Gauge)
90	10	4.5 to 11.0
	15	6.5 to 17.0
	20	8.5 to 30.5
100	10	0 to 8.5
	15	8.0 to 17.5
	20	13.0 to 25.5

(d) So far as the committee is aware, the present I. C. C. method has served very satisfactorily as far as transportation hazard is concerned. Consequently, from a safety viewpoint there is comparatively little need of change under existing

marketing conditions since relatively stable natural gasolines are customarily shipped.

(e) Other vapor pressure methods are being considered because the industry feels that a better vapor pressure method will be of advantage in the transportation and marketing of natural gasoline. The committee feels that the industry should not use any testing method which is admittedly inaccurate. The committee also feels that the adoption of an exact vapor pressure method will serve to better define natural gasoline and thus extend markets.

(f) Many vapor pressure methods have been proposed. From the data at hand, the committee has concluded that the Beistle-Prather and Reid methods are the only ones of those proposed which are practicable and which yield results reasonably approximating true vapor pressures.

Attention is called to Figure 13, P. 118, and Figure 15, P. 120, of the December, 1927, A. P. I. Proceedings in which Reid and Beistle-Prather vapor pressure values are compared with "true" vapor pressure values. A very close agreement is noted. The Reid vapor pressure values are slightly lower than "true" vapor pressures while the Beistle-Prather vapor pressure values are slightly higher than "true" vapor pressure values. Both methods when applied to pure liquids yield results which approximate accepted vapor pressure data for those liquids.

Having reached the conclusion that the Beistle-Prather and Reid vapor pressure methods yield results which closely approximate "true" vapor pressures a large number of data become available for comparing Engler distillation characteristics and the present I. C. C. vapor tension values with "true" vapor pressure values. For example, in considering distillation loss as an index of "true" vapor pressure it was thus ascertained that natural gasoline having a distillation loss of eleven per cent could have "true" vapor pressures as high as 18 lbs. per sq. in gauge at 90 F. and 23 lbs per sq. in. gauge at 100° F. Such data serve to materially increase the ranges of "true" vapor pressure noted in the two tables previously presented and further emphasize the conclusions made in regard to them.

(g) Comparing the Beistle-Prather vapor pressure method with the Reid vapor pressure method, the former gives more information regarding the samples tested than the latter. For example, the Beistle-Prather method gives some information as to the quantity and quality of the foreign and fixed gases and consequently affords some indication as to the "actual" vapor pressure of the gasoline with all its contaminants. Data from Beistle-Prather vapor pressure tests, in the hands of a person capable of interpreting them, might prove valuable. The only information obtainable from the Reid vapor pressure tests is the "true" vapor pressure. The Beistle-Prather method is more complicated in its manipulation, a single test takes more time, the apparatus is more costly, and there is a slightly greater chance for leaks than is the case with the Reid apparatus.

(h) Both the Beistle-Prather and Reid vapor pressure methods would be comparatively easy to put into practice.

(i) Because of its greater simplicity, the committee believes the Reid method would be a more reliable test in the hands of loading rack men than the Beistle-Prather method.

(j) It is recommended by the committee that either the Reid or the Beistle-Prather method be adopted by the N. G. A. A. as the method for determining the vapor pressure of natural gasoline. The committee prefers the Reid method, but feels that the Beistle-Prather method would be a very satisfactory alternative.

(k) The temperature of the bath should be the same throughout the year. The temperature recommended by the committee is 90 degrees F. Vapor pressures for any other temperatures can be calculated with a reasonable degree of accuracy from the 90 degree value.

The tentative standard vapor pressure testing method adopted, to become effective September 1, 1928, is given below in detail.

Apparatus

The apparatus and method of procedure is essentially the same as proposed by Walter Reid, which apparatus consists of a bomb, a pressure gauge and a water bath.

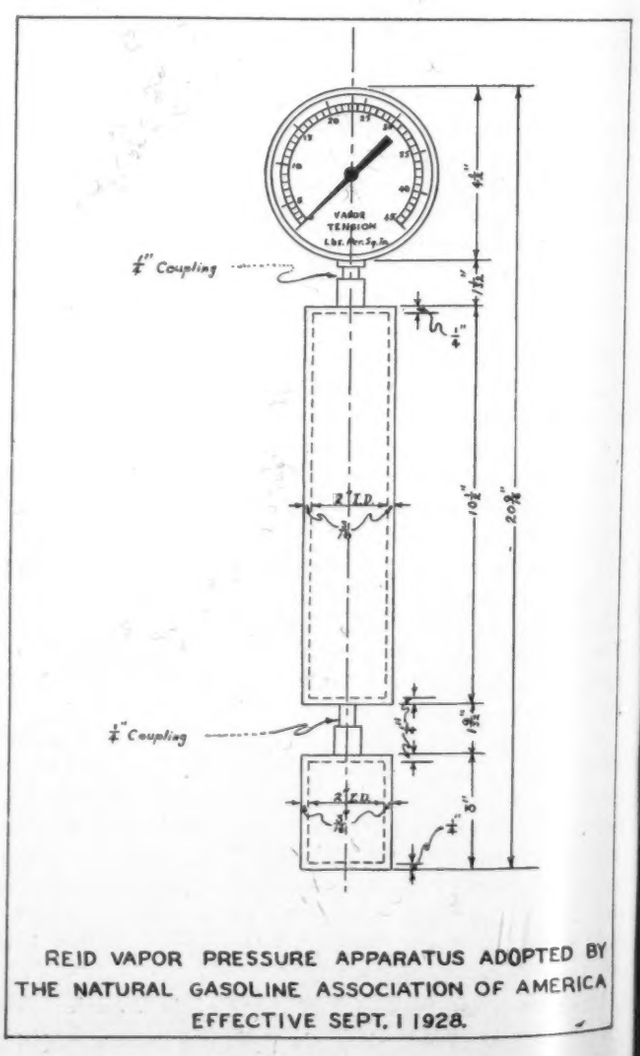
(*a*) *The Bomb*—The bomb consists of two parts. The first, which is the air bomb, is made of a piece of 2-inch standard line pipe 10 inches long, with welded ends having a ¼-inch collar welded in the center of one end, and a ¼-inch nipple 1 inch long welded in the center of the other end. This bomb *must* drain completely from either end when held in a vertical position.

The second part, the gasoline bomb, consists of a piece of 2-inch standard line pipe 2½ inches long with welded ends having a ¼-inch collar welded in the center of one end, the other end being completely closed.

This apparatus is shown in the figure.

(*b*) *Pressure Gauge*—The pressure gauge shall be an approved make of vapor tension or test gauge, with a diameter of 3 or 4½inches, and scaled for 30 or 45 pounds pressure. Gauges shall be checked frequently and adjusted if found to be in error.

(*c*) *Water Bath*—The water gath shall be of such dimensions that the bomb may be immersed to the bottom of the pressure



REID VAPOR PRESSURE APPARATUS ADOPTED BY THE NATURAL GASOLINE ASSOCIATION OF AMERICA EFFECTIVE SEPT. 1 1928.

gauge. Means for maintaining a constant bath temperature of 90 degrees F. should be provided.

Procedure

(*a*) *Purging the Air Bomb*—Prior to each test the air bomb must be thoroughly purged of any gasoline or gasoline vapors that may remain from a previous test. This is best accomplished by filling the air bomb, from which the gauge has been removed, with hot water, then allowing the bomb to drain. This procedure should be repeated several times. The air bomb is then suspended in the shade on a rack in a vertical position. The bomb should remain on the rack for at least one hour.

Where a large number of determinations are to be made, several air bombs should be provided for each gasoline bomb.

Just prior to the test the pressure gauge is screwed tightly into the air bomb, using a little shellac or powdered lead on the threads of the gauge to make a tight connection.

(*b*) *Filling the Gasoline Bomb*—Before each test the gasoline bomb is rinsed with water. This water is poured out and the bomb allowed to drain for one minute.

The bomb is immersed in a tank of the material to be tested and allowed to fill while being raised and lowered through the liquid. This liquid is used to thoroughly wash the bomb and is then emptied. The bomb is refilled in the same manner, care being taken to see that it is completely full of the liquid. The sample is now ready for the test.

Should it be necessary to transfer the sample to the bomb by means of pouring, both the liquid and the bomb must be cold enough so that no appreciable change in composition of the liquid occurs during the operation. The bomb shall be completely full of the liquid when the test is run. Table 1 gives the temperatures at which gasolines of several vapor pressures should be held when pouring.

TABLE 1

Temperature to Which Gasolines Should Be Cooled Before Pouring.

Vapor Pressure @ 90°F. Lbs. per Sq. In. Abs.	Temperature of Sample When Pouring
9 or under	32 - 40°F.
9 - 12	15 - 20°F.
12 - 16	10 - 15°F.

(*c*) *Conducting Test*—The temperature of the air in the air bomb is recorded. The air bomb with the gauge in place is then immediately attached tightly to the gasoline bomb. The apparatus is turned upside down several times to allow gasoline to run into the air chamber. It is then immersed in a water bath held constant at 90 degrees F. At the end of five minutes the bomb is removed from the bath, inverted several times and shaken vigorously. It is then replaced in the bath, and as soon as the gauge reading becomes constant, the pressure is recorded. This will usually require from five to ten minutes.

(*d*) *Calculating Results*—In order to calculate the vapor pressure of the gasoline, the final pressure recorded above must be corrected for the change in the pressure of the air in the bomb due to change in temperature. This is done by adding to or subtracting from the observed pressure the correction given in Table 2, opposite the air bomb temperature. Since the pressure of the air in the air bomb was zero pounds gauge when attached to the gasoline bomb, any pressure shown on the gauge at the end of the test must have been due to the pressure of the gasoline vapors and the change in the air pressure. For this reason, the gauge pressure when corrected for the change of the air pressure is the vapor pressure of the gasoline in pounds per square inch absolute. For example, the observed gauge pressure is 15.0 pounds per square inch and the air bomb temperature is 76 degrees F. Reference to Table 2 will show the correction for this air bomb temperature to be 0.4 pounds per square inch. Subtracting this correction from the observed gauge pressure of 15.0 pounds will give 14.6 pounds per square inch absolute as the vapor pressure of the gasoline.

TABLE 2

Correction Factors for Expansion of Air.
(Barometric Pressure 14.4 Lbs. Sq. In.)

Air Bomb Temp. °F.	Correction	Air Bomb Temp. °F.	Correction
30	Subtract 1.8	72	Subtract 0.5
32	1.7	74	0.4
34	1.6	76	0.4
36	1.6	78	0.3
38	1.5	80	0.3
40	1.4	82	0.2
42	1.4	84	0.2
44	1.3	86	0.1
46	1.3	88	0.1
48	1.2	90	0.0
50	1.1	92	Add 0.1
52	1.1	94	0.1
54	1.0	96	0.2
56	1.0	98	0.2
58	0.9	100	0.3
60	0.8	102	0.3
62	0.8	104	0.4
64	0.7	106	0.4
66	0.7	108	0.5
68	0.6	110	0.5
70	0.5		

Figures given in table to be subtracted from or added to the observed reading to give vapor pressure of gasoline in pounds per square inch absolute at 90 degrees F.

The above values were calculated from the formula:

$$\text{Increase in air pressure} = \frac{\text{Barometric pressure} \times (90 - \text{Air Bomb Temp.})}{460 + \text{air bomb temperature}}$$

New Gasoline Company Organized

Shreveport, Louisiana.—Combining their interests, the Root Refineries, Inc., and F. C. Henderson have organized the Rainbow Gasoline Company; and the new company is operating a gasoline plant in Union County, South Arkansas. The plant uses 10,000,000 feet of gas daily, putting out 20,000 gallons of finished gasoline per day. The supply of gas for the plant comes from wells in the Champagnolle field. The cost of the plant was around $250,000, and the cost will run still higher, as additional compressors are being installed.

The Rainbow company was organized late in July, with 100 no par value shares divided equally between F. C. Henderson and the Root Refineries, Inc.; the latter concern is owned in large part by D. S. Brooks and D. P. Hamilton of Shreveport.

Officers of the Rainbow Gasoline Corporation are: D. S. Brooks, president; D. P. Hamilton, vice president; F. C. Hall, secretary; and F. C. Henderson, treasurer.

The new gasoline plant is located on the H. L. Hunt 120-acre lease, Gregory farm, comprising the southwest of the southeast and the southeast of the southwest of Section 10-17-14, and the northeast of the northwest of Section 15-17-14.

Beaumont, Texas.—The Atlantic Refining Company is planning the construction of a $500,000 refinery at Atreco on the Neches River near here; and has proposed to build a pipe line of 30,000 barrels daily capacity from Midland, Texas, to the refinery site at a cost of around $2,000,000.

Gas and Gasoline Analysis as Applied To Plant Control

By J. C. BURKS
O. C. Field Gasoline Corporation

Read before the meeting of the California Natural Gasoline Association, August 9, 1928.

WE are interested in producing natural gasoline of such specifications that the refiner can include it in motor fuel to the extent that a maximum return to the natural gasoline manufacturer is realized. This will be accomplished through the production of a relatively large volume of low distillation recovery gasoline on one price scale, or through the production of a smaller volume of high distillation recovery gasoline on a higher price scale, or on some intermediate basis. Whatever the basis decided upon, however, we are interested primarily in producing all of the gasoline meeting this specification contained in the gas treated. Furthermore, there is no denying the fact that we are confronted with economic conditions that are likely to continue indefinitely, both from a standpoint of overproduction and the encroachment of cracking processes. It is obvious that plant operating methods will have to keep pace with these conditions and that methods of plant control that have been generally used, particularly charcoal tests, are unsatisfactory and inadequate.

As the average efficiency of gasoline plants generally has increased, the percentage of the lighter constituents included in the product increased greatly, resulting in more stringent specifications being written by the refiner until we now generally accept 90 per cent. distillation recovery as a proper recovery for a good grade of natural gasoline. It is not the purpose of this paper to discuss the relative merits of natural gasolines as related to gravity, distillation recovery, vapor pressure, or volatility, but to take conditions that are now imposed on the average operator and attempt to show how he may be assisted in producing the maximum gallonage meeting his particular sales specifications. I believe the desired results can be attained only through accurate knowledge of what his raw material consists, and the exact composition of his products.

Theoretically the desired operating condition would be the removal of all of the butanes and heavier fractions from the wet gas with a minimum amount of propane and lighter, the elimination of all of the propane and lighter from the condensed make and the elimination of just sufficient butanes to give the required distillation recovery. As the production of propane free natural gasoline is accomplished through a substantial loss of heavier constituents (even in the best regulated stabilizing units) the usual plant operating balance will be found where small percentages of propane remain in the finished product, with a correspondingly greater net production for a given recovery specification. The other extreme

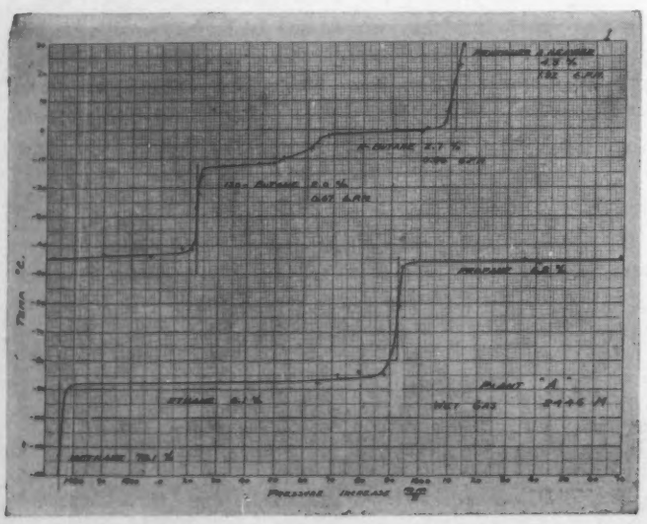


Figure 1.

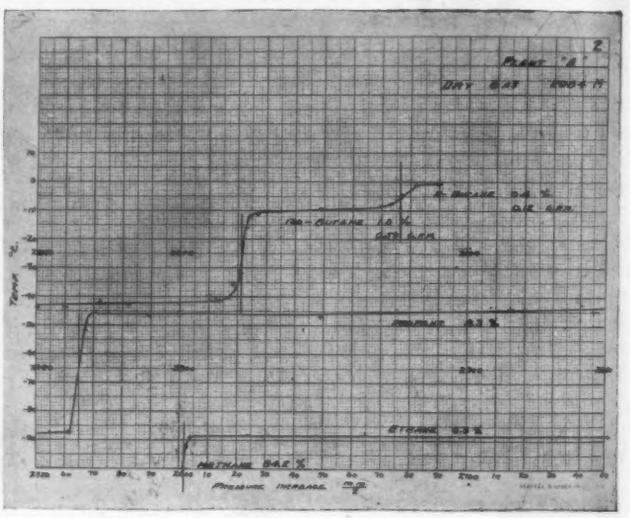


Figure 2.

is found in plants that are leaving substantial amounts of butane in their residual absorber gases and are having difficulty meeting distillation recovery requirements on the gasoline they are making. Between these two extreme cases lie innumerable plant operating conditions, all confronted, however, with the same question: How many gallons of specification gasoline are contained in the gas available for treatment and what are the plant conditions most suitable for its recovery. In other words, a plant will lose, we will say, 10 per cent. of its production in increasing its distillation recovery from 85 to 90 per cent. What part of that loss was due to elimination of propane? How much butane was lost along with it? Could larger amounts of butane be included in the make if still more propane was eliminated, and if so how much? Etc.

To assist us in a few of these problems, we started experimenting last winter with an analysis apparatus based on the principle of cold fractionation for determining the hydrocarbon components of gasoline and gases. The apparatus consists of a carefully packed fractionating column, the upper or condensing portion of which is controlled by a refrigerant. The different constituents are distilled off at constant temperatures and measured as vapor in a large receiver by means of pressure increases.

Typical Analyses

We will now consider a few typical analyses and see how they may be interpreted: Figure 1 shows an analysis of wet gas entering Plant No. A, a 30 pound 32 degree charcoal determination as specified by Bulletin No. TS 121, of which indicated 3.10 GPM. We find that this gas contained 78.1 volume per cent. of methane; 6.1 per cent. ethane; 6.8 per cent propane; 2.0 per cent. or 0.67 GPM iso-butane; 2.7 per cent. or 0.86 GPM N-butane; and 4.3 per cent. or 1.92 GPM pentanes and higher.

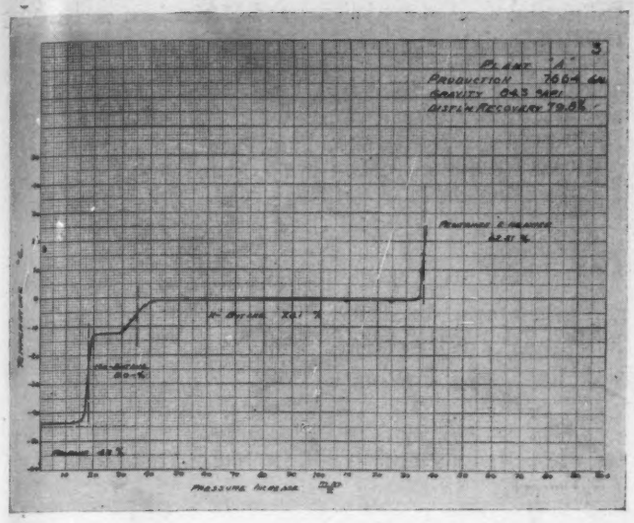


Figure 3.

The question now arises, is the analysis accurate? Doctor Brown made a statement at Tulsa that his apparatus was accurate to within one-tenth of a per cent. in testing motor fuels. We do not find such accuracy necessary, however, in plant control work, nor do we imagine that we are getting results anywhere near this accuracy; but through refractionation of the separated constituents we are certain that the apparatus is more accurate in determining the components of gasoline and gases that are the other yardsticks by which plant efficiency is determined. We do not consider a master meter on a plant inaccurate, for instance, if it is 1 per cent. off. Gasoline measusements at different temperatures are likely to be one-half of a per cent. off, so for practical purposes we consider the accuracy in determining the hydrocarbon components of gasoline and gases by this method is more accurate than the methods used to determine other factors in the plant equation.

Analysis 2 shown here as Figure 2 is the dry gas leaving the absorber of the same Plant A. Charcoal analysis showed 0.33 GPM. The gas contained 84.2 volume per cent. methane; 5.5 per cent. of ethane; 8.3 per cent. propane; 1.8 per cent. or 0.6 GPM iso-butane; and 0.4 per cent or 0.12 GPM N-butane.

Analysis 3, Figure 3, is a sample from the run tank of this same plant No. A; showing 4.3 per cent. propane; 5.0 per cent. iso-butane; 28.2 per cent. N-butane; and 62.5 per cent. pentanes and higher. Gravity of this product was 84.3 degrees; distillation recovery 79.8 per cent. You will note from these analyses that it is entirely possible to fractionate mixtures of the lower

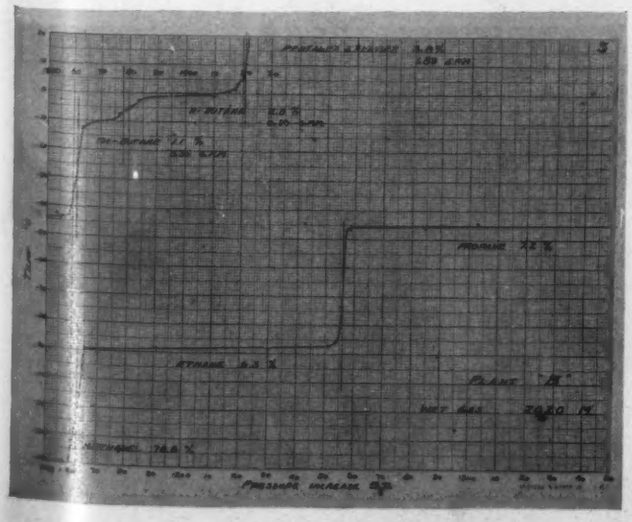


Figure 5.

paraffin hydrocarbon series into their components with rather sharp cuts, with the possible exceptions of iso and normal butane. Even in this case where the difference in boiling points at atmospheric pressure is only about 12 degrees C., a very sharp cut can be made if time is allowed for it. However, we find that the same result is obtained in running more rapidly and extrapolating the curve as shown here as by taking a considerable longer time and obtaining a sharper break.

We will now consider a somewhat similar case except that

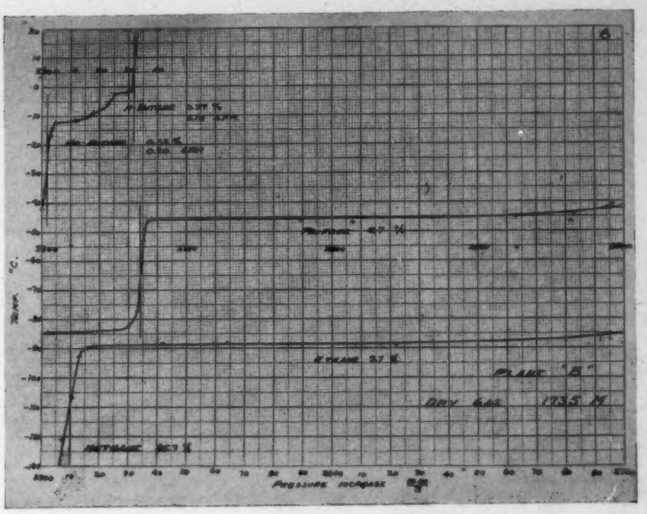


Figure 6.

here a stabilizing unit is involved, although operating under extremely poor conditions.

Figure 5 shows an analysis of wet gas entering Plant No. B. A 30 pound 30 degree charcoal analysis showed 3.0 GPM Methane 78.8 volume per cent; ethane 6.3 per cent; propane 7.2 per cent; iso-butane 1.1 per cent or 0.35 GPM; N-butane 2.8 per cent or 0.90 GPM; pentanes and higher 3.8 per cent or 1.89 GPM. Total of iso-butane and heavier 3.14 GPM.

Figure 6 is analysis of dry gas from absorber. Methane 85.7 per cent; ethane 7.7 per cent; propane 5.7 per cent; iso-

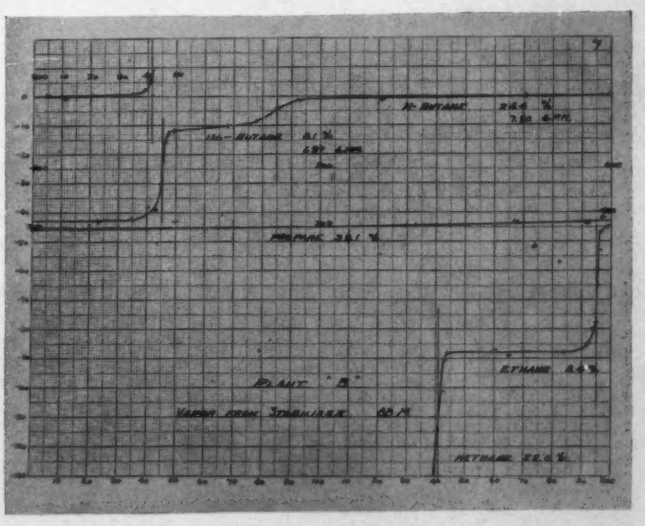


Figure 7.

butane 0.20 GPM; N-butane 0.12 GPM. Charcoal analysis 0.30 GPM at 30 pounds, 32 degrees.

Figure 7 is vapor from stabilizing column showing methane content 22.0 per cent; ethane 8.4 per cent; propane 39.1 per cent; iso-butane 6.1 per cent or 1.97 GPM; and N-butane 24.4 per cent or 7.90 GPM.

Obviously this column is in a bad way, particularly when we consider that the production still contains 2.8 per cent. propane. (Figure 8.) The rest of the analyses shows iso-butane 2.4 per cent; N-butane 17.1 per cent; pentanes and higher 77.7

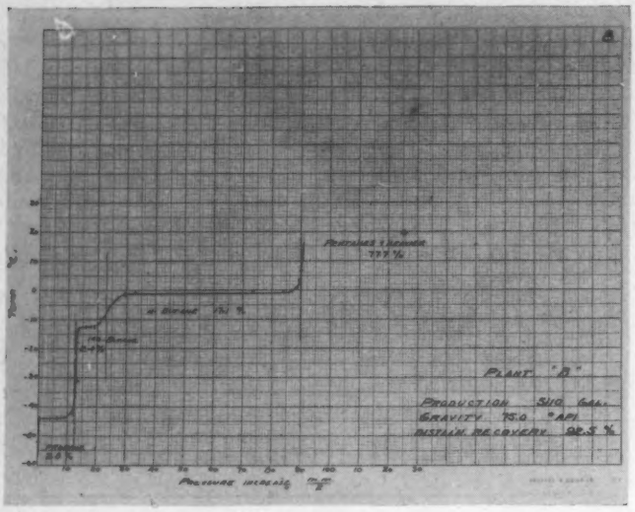


Figure 8.

per cent. Gravity of the product was 75.0 degrees; distillation recovery 92.5 per cent.

If we now take the results of these analyses and the amount of gas entering and leaving the plant as well as production, we may arrive at a plant balance as shown in Figure 9. Iso-butane and heavier are shown in gallons. Propane and lighter as M cubic-feet. You can see that the sum of the different constituents contained in the production, the dry gas and stabilizer vapor are shown just between the diagonal double lines and you can see the check or lack of it, with the constituents entering the plant. Considering the fact that these samples were taken over a two-hour period and that gas and gasoline volumes were taken over 24 hours, a closer check was not anticipated. Anyhow, we have 1,592 M cubic feet of methane in the incoming gas, against 1,504 accounted for in the plant. 127 M cubic feet ethane against 141; 146 M cybic fgeet propane against 138; 707 gallons iso-butane against 643; 1820 gallons N-butane against 1,777; and 3,820 gallons pentane and higher against 3,970.

We will come back to this plant balance in a moment and see what effect better operating conditions would have on the amount of gasoline made and its specifications.

We will now consider a stabilizing unit producing approximately 20,000 gallons per 24 hours, of 81 gravity, 90 recovery gasoline. Figure 10 shows an analysis of gasoline from run tank fed directly from column; rate of make 20,000 gallons; propane 2.8 percent; iso-butane 3.0 percent; N-butane 22.6 percent; pentane and higher 70.9 percent.

Figure 11 is residue vapor leaving stabilizing column in the amount of 175 M cubic feet; indicating 13.3 percent methane;

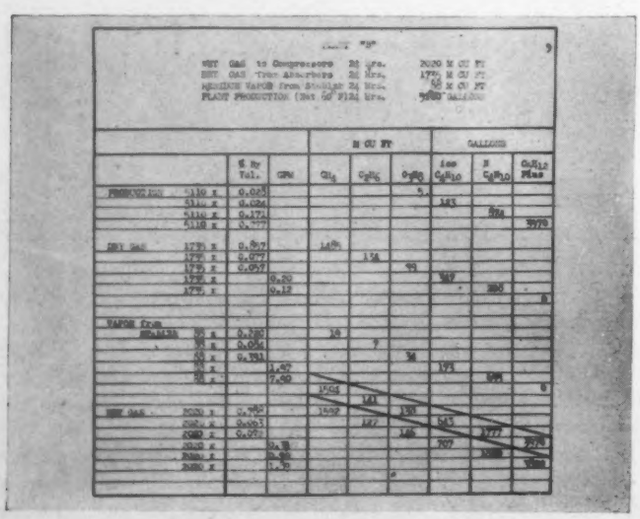


Figure 9.

12.2 percent ethane; 49.7 percent or 13.6 GPM propane; 9.7 percent or 3.14 GPM iso-butane; and 15.1 percent or 4.8 GPM N-butane.

Figure 12 shows vapor leaving the run tank which was maintained at 5 pounds pauge pressure, amounting to 38 M cubic feet consisting of:

24.2 volume percent or 6. 6. GPM Propane.
8.1 volume percent or 2.6 GPM Iso-butane.
52.2 volume percent or 16.9 GPM N-butane.
15.5 volume percent or 6.5 GPM Pentane plus.

A compilation of these analyses resolves the products of the unit into amounts as shown in Figure 13, and it is noted that

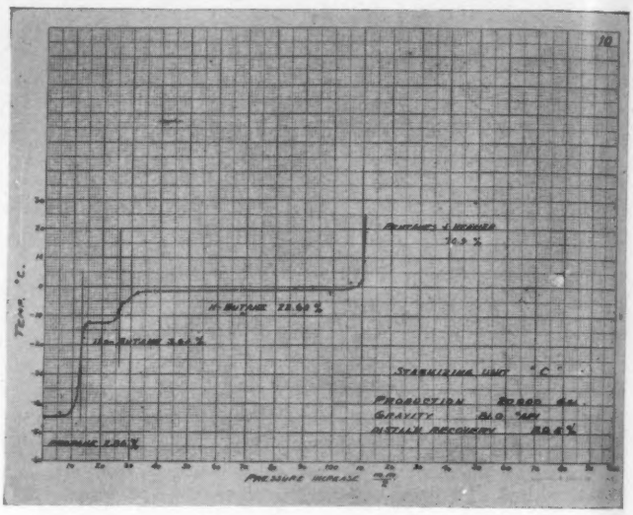


Figure 10.

if methane and ethane are converted to their liquid equivilents that we have accounted for

235 gallons Methane
364 gallons Ethane
2816 gallons Propane
1276 gallons Iso-butane
5870 gallons N-butane
14427 gallons Pentane plus.

From these figures we can calculate that the run down stream or entering feed to the column had the composition:

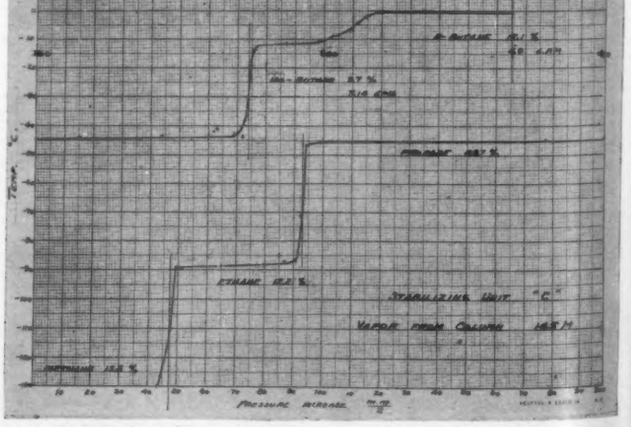


Figure 11.

0.9 percent Methane
1.4 percent Ethane
11.3 percent Propane
5.10 percent Iso-butane
23.5 percent N-butane
57.8 percent Pentane plus.

Now, as a means of interpreting these analyses still further, let us consider the diagram in Figrue 14. You are undoubtedly familiar with this method of three dimensional plotting. Obvi-

ously if we take an equilatoral triangle whose perpendicular equals unity, the sum of the perpendiculars from any point in the triangle to the three bases equals unity. Therefore, if we allow distances from one base to equal percent of propane, distances from another base to equal percent of total butanes, and distances from the third base to equal percent of pentenes and higher, we can take the analyses of gasoline samples and at least get them down on paper. The scattered points you see in this diagram are a few such analyses. For most gasolines, these points will fall in the lower right of the triangle so we will enlarge the scale and include this area only.

We have taken the results of all the liquid analyses we have run so far, and located them on diagram as shown in Figure

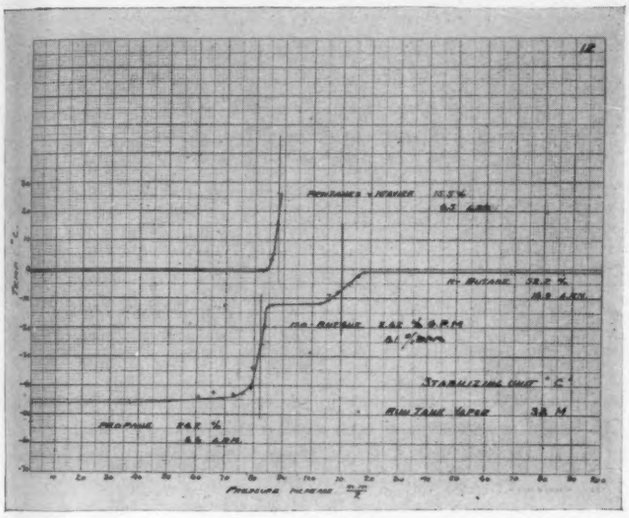


Figure 12.

15, noting on each point the ASTM distillation recovery of the sample. It would appear that until we get analyses and distillation recoveries that disprove it, it should be possible to appropriate the distillation recovery of a gasoline of a given percentage composition of propane, total butanes, and pentanes plus, if said distillation recovery lies roughly between 78 and 95 degrees. We are assuming of course that the composition of the pentanes and heavier fraction remains about constant and that differences in the amount of iso-butane as related to normal butane will not substantially affect distillation recovery.

I believe that we can make our first assumption safely when we consider that the distillation recovery of the usual pen-

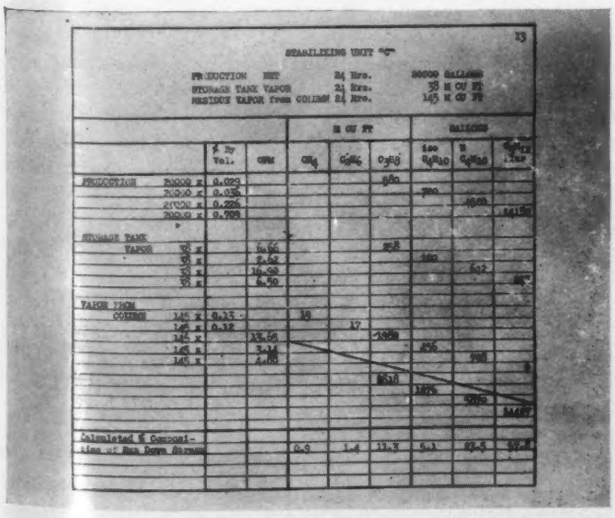


Figure 13.

tanes and heavier fraction of a natural gasoline is nominally 100 percent and that even appreciable fluctuation in the

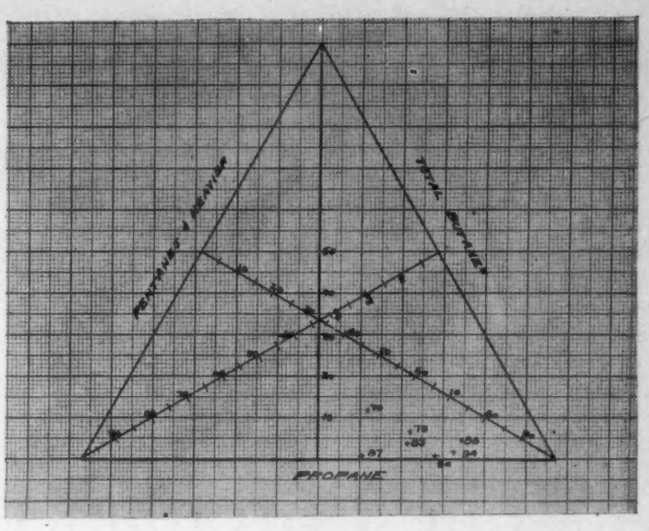


Figure 14.

percentage of pentane contained therein would not affect the distillation recovery of the sample. As far as iso and normal butane are concerned, their differences in relation to each other undoubtedly account for some of the obvious discrepencies on the composition diagram. A suggested remedy is the conversion of iso-butane to its N-butane equivalent in respect to distillation loss, by some approximate method. Nevertheless, from a plant operating standpoint, we can use this tendency toward a relationship between percentage composition and distillation recovery to advantage.

For instance, let us again consider plant B with the stabilizing unit so-called. We found that 208 gallons of N-butane were lost in the residual gas from absorber and 695 gallons from residual vapor from the stabilizing column. If we now change operating conditions so that all of this normal butane is recovered, we should have 6013 gallons of a product consisting of 2.4 per cent. propane, 31.6 per cent. total butanes, and 66.0 per cent. pentanes and higher. Referring this analyses to our diagram, we would anticipate a distillation recovery of approximately 88.0 per cent. If we now make the stabilizing column perform so that the propane is cut down to 0.5 of one per cent., we would have 5900 gallons of gasoline of approximately 90 per cent. distillation recovery. It is obvious that there are any number of possible combinations of propane and total butanes here in which the plant could be made to produce 90 per cent. recovery gasoline and with these data at hand an operator could determine the optimum set of conditions for his particular plant.

In a similar way, we may consider stabilizing unit C. Here,

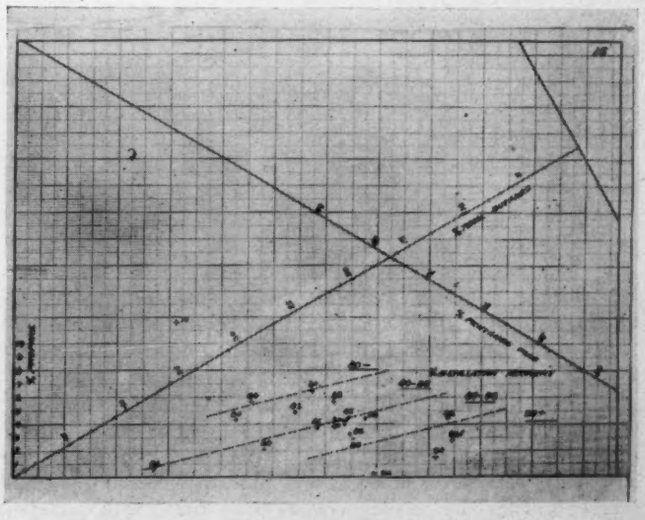


Figure 15.

we are losing 1164 gallons of total butanes in vapor from column, which, if included in the make without any other change would give us 21,164 gallons consisting of 2.7 per cent. propane; 30.3 per cent. total butanes; and 67.0 per cent. pentanes and higher. From the diagram we would expect a distillation recovery of between 87 and 88 per cent. However, in reducing the propane content to 0.5 per cent., we have 20,688 gallons of product that is safely within the 90 per cent. recovery requirement. On the other hand, if it was necessary to produce 95 per cent. recovery gasoline from this unit, assuming that 0.5 per cent. propane was the lowest practical limit at which to operate, we would have to limit our total butane to about 21 per cent., cutting our production from 20,688 gallons of 90 per cent. recovery make to about 18,000 gallons of 95 per cent., a loss in production of approximately 13 per cent. Under these conditions it is interesting to note that the gravity of the product would be reduced from 83.5 A.P.I., to about 79 A.P.I.

Assuming that 0.5 per cent of propane is a practical working basis, it would appear that for a 90 per cent recovery product the total butanes will have to be limited to approximately 32 per cent.

I might add that since these plates were made we have established a dozen or so more points on this diagram that further bear it out, but I must emphasize he fact that these data only indicate a general tendency toward a relationship that is obviously limited to the accuracy of our ASTM distillation itself.

In conclusion I would say that I believe that future plant control will be along lines we have discussed here, and that future research should be directed toward a more definite relationship between percentage composition and sales specifications.

The Foreman's Part in Accident Prevention

By C. W. PRICE

Director, Training Course for Safety Engineers, New York University

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IT is a commonplace and yet peculiarly applicable to accident prevention that the attitude of the employe depends absolutely upon the attitude of the foreman.

That is why the foreman's interest in accident prevention must be so sincere and unmistakable that none of his men will doubt for a minute what he is willing to do to protect them and what he expects them to do.

Pretense accomplishes nothing in a safety campaign. The old foreman's alibi to his men: "This is a lot of nonsense but the boss says it's got to be done," defeats its very purpose. Either the foreman believes in safety or he doesn't and his men will soon find him out. Not just ordinary interest, but special interest and enthusiasm are indispensible for a foreman to make his men really believe in safety and give it a place in their daily work.

The foreman must believe in accident prevention as a good business proposition, not merely as a bit of humanitarianism, if the men under him are to make honest efforts to avoid accidents. He must believe it pays good dividends—that it is an indispensible part of efficiency. If he doesn't, he will never quite put safety on the map in his department and give it a vital place in the day's work. It will always be a side issue, "kind hearted but not quite hard headed business," and his men will all know it.

Let's enumerate the few specific things a foreman must do to make his department safe:

1. It goes without saying that he must cooperate with the management in the installation of mechanical guards and other safety equipment and he must make himself responsible for seeing that these guards are kept in good repair and are used by his workmen.

2. Because good housekeeping plays a very important part in accident prevention, the foreman should make it his business to see that the floors are kept free from obstructions over which men may stumble, that materials are piled safely, and that ladders, stairways and tools are kept in good repair.

3. It is of the utmost importance that the foreman personally introduce the new man to his job to which he is adapted; that he carefully instruct the new employe as regards the hazards of his job and how to perform the work safely; and that he should take the opportunity with each new employe to impress upon him the interest which the officers and the employes are taking in accident prevention and what he, the foreman, expects of the new employe.

4. When any employe is found violating a safety rule, the foreman must not only correct him but should do so in such a way as to win the intelligent interest and cooperation of the employe. In other words, he should make safety attractive, not merely mandatory.

5. The foreman should not only make regular inspections of his department but should exercise constant watchfulness to weed out dangerous conditions and dangerous practices.

6. While discipline occupies only a minor place in a safety campaign yet there are occasions when discipline is necessary. In cases where there has been wilfull and flagrant violation of safety rules or safe practices, it is often necessary to discharge an employe. In the majority of cases, however, a layoff for a few days or several weeks proves effective in correcting the condition.

Foremen should never forget that in the end the whole success of accident prevention depends on whether workmen really believe in it, in whether they are really interested, and in whether they appreciate its value to them personally. In any disciplinary measure, therefore, the foremen should be careful to avoid breaking down this spirit or antagonizing whole groups of employes. Above all, foremen and managers should never announce penalties for violation of safety rules unless they intend to enforce them, for such a practice—still common in hundreds of plants—does more to breed disregard and contempt for safety work than anything else.

It should be remembered by management as well as by foremen that in the last analysis the foreman is the key man in any safety program. To most factory employes, the foreman is "the boss," in fact the entire company. Certainly to them the company is gaged by the calibre of the foreman. The foreman, alone is in constant touch with the workmen, the men and women involved in most industrial accidents. Management, therefore, can do nothing better for the promotion of safety than to sell the idea thoroughly to its foremen, keep them sold and interested, and as deeply concerned with safety records as with production records. Those plants are most successful in accident prevention in which foremen are held as closely to account for loss of time and dollars through preventable accidents as for loss of material and orders through preventable spoilage and delay in production.

Heat Conservation Paramount With Pure Oil at Heath

By GEORGE REID
Associate Editor

THE Heath refinery of the Pure Oil Company near Newark, Ohio presents a pleasing picture by virtue of its excellently landscaped and well kept front yard, artistically arranged flower gardens surround its main buildings, which are set off by bright aluminum coated tanks and its numerous buildings of brick and concrete, topped by red tile roofs. In addition to the pleasing appearance secured by constant care and cleanliness, there is incorporated in the design and operation of the refinery proper many interesting features of economy and efficiency. Space here does not permit of treating both sides of the picture and the excellent results secured by welfare organizations, safety first groups, foreman's councils and so forth, will have to give way for the discussion dealing with interesting items of plant operation.

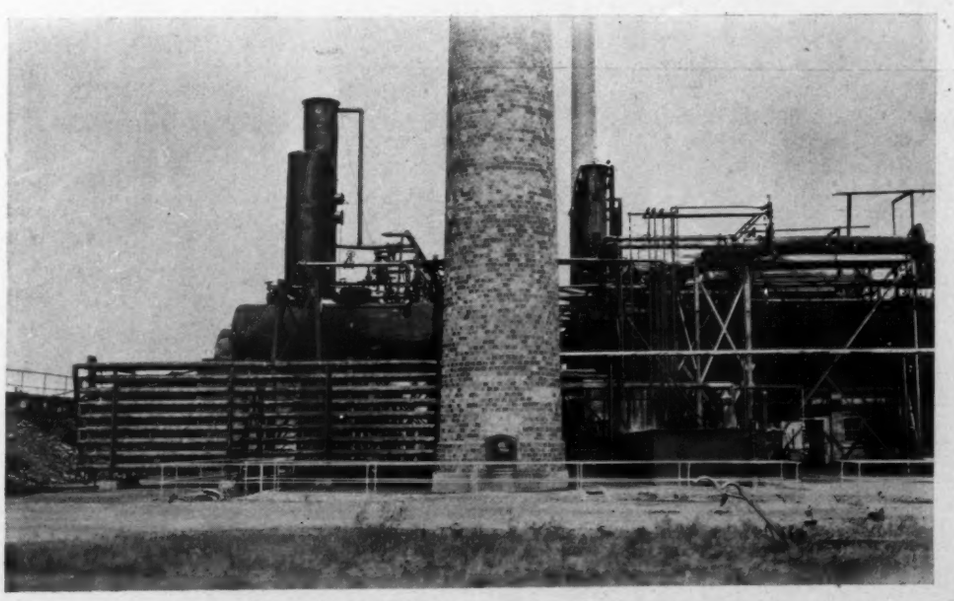
The crude refining equipment at the Heath plant consists of 13 shell stills arranged in two batteries for skimming work and redistillations. Two older type Cross units of 750 barrels capacity and two of the new large type Cross units of 2,000 barrels rated capacity, which are operated much above that figure, comprise the cracking equipment. In addition a large unit of the Gyro vapor phase cracking system shortly will be in operation. This Gyro unit will charge crude oil direct to the assembly of equipment and skimming and cracking will be performed in the same pass through. Throughout all of the departments where oil is processed company engineers have given much attention and study to heat conservation and better fuel economy.

At the skimming battery, crude oil entering the equipment is preheated to 400 F. before it enters the first still. At this battery the cold oil is charged through four vapor heat exchangers which are installed on the vapor lines leading from two kerosene and two gas oil stills to the condenser boxes. These exchangers are of company design and consist of 90 one-inch tubes, each, incorporating an expansion head in such a manner that expansion and contraction are adequately cared for. Passing from the four vapor heat exchangers the crude is then conducted to an exchanger of conventional type, also company designed, where it absorbs heat from the hot residuum leaving the last still in the series.

Heat Conservation

From this last exchanger where the preheat temperature reaches 400 F. the crude is split into two streams and passes into two large drums surmounted by small towers, called evaporators. At this point two streams of light gasoline are removed to condenser boxes, and through the receiving house. Sixty per cent. of the total available gasoline, by skimming, is removed in this manner, before the crude reaches No. 1 shell still. The advantage of having two evaporators and of removing two streams in this manner lies in the fact that in the event of failure of any of the evaporator, tower, or condenser equipment, the still operation need not be interrupted while repairs are made.

The vapor heat exchanger installation effects not only greater yield of gasoline through exchange, but materially reduces the fuel requirements for distillation work. They also reduce the water needs of the condenser boxes and at the same time give cooler streams, and keep the boxes cooler. The reason for placing the exchangers on the heavier stream vapor lines is obvious. The skimming bat-



At the left the two evaporator drums are shown and at the right, above the condenser boxes, are two of the vapor heat exchangers as described in the accompanying article.

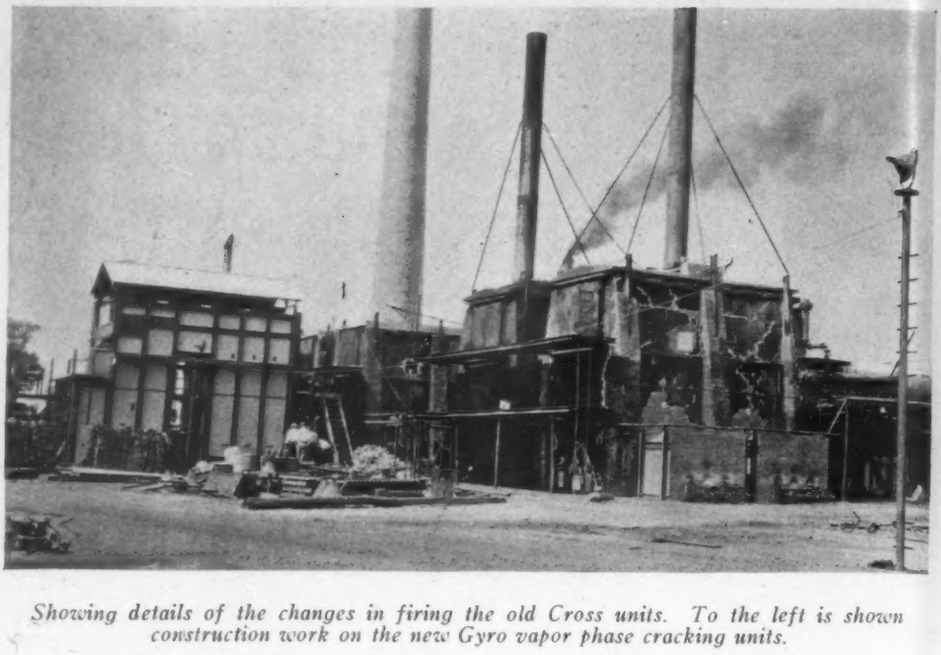
tery produces the usual fractions of gasoline, kerosene, and gas oil. Both the gas oil and fuel oil are utilized as cracking stock for the four Cross units.

The plant is independent of outside utility services in so far as it produces its own electricity and water. Coal is used in the boiler house as fuel. Gas is used in some departments when available from the cracking units, and oil fires are found for some of the processing. A steam driven Corliss engine, 350 horsepower is employed to generate electricity for power and lights and to drive compressors for the supply of compressed air needed about the plant, and to belt drive the large compressor acting as the present gasoline recovery unit. All generators and compressors are belt driven from a common shaft which is driven by a Corliss engine. A Ridgeway steam engine, 265 horsepower, is used as a stand-by unit. Two Ingersoll-Rand and one Laidlaw compressor are installed for supplying compressed air to the centralized air storage tank. A Laidlaw compressor size 16 by 8 by 16, compresses gases and vapors from all parts of the plant for gasoline recovery purposes. The power house consists of four 375 horsepower boilers, coal fired.

A new waste gas gasoline recovery plant is being installed which will be double the capacity of the present plant. It will consist of two Bessemer compressors shipped to Heath from one of the Pure's Seminole lease, and one Ingersoll-Rand machine, and the capacity of the recovery plant will be increased to 4000 gallons of gasoline daily.

Electric Plant

The electricity generated by the Crocker-Wheeler generators is taken to all parts of the refinery and used for both power and lighting purposes, and also provides light for the large employees' camp near the plant. Because of the abundant supply of current the majority of the pumping is done by electrically driven pumps. The main pump house, for example consists of seven Goulds triplex pumps, all driven by a single 50 horsepower Crocker-Wheeler motor by means of a common shaft and gears. This installation has been in service nine years. The pumps are located on one side of the house, as shown in one of the accompanying illustrations, with all of the manifolds placed on the other side of the building directly opposite the pumps.

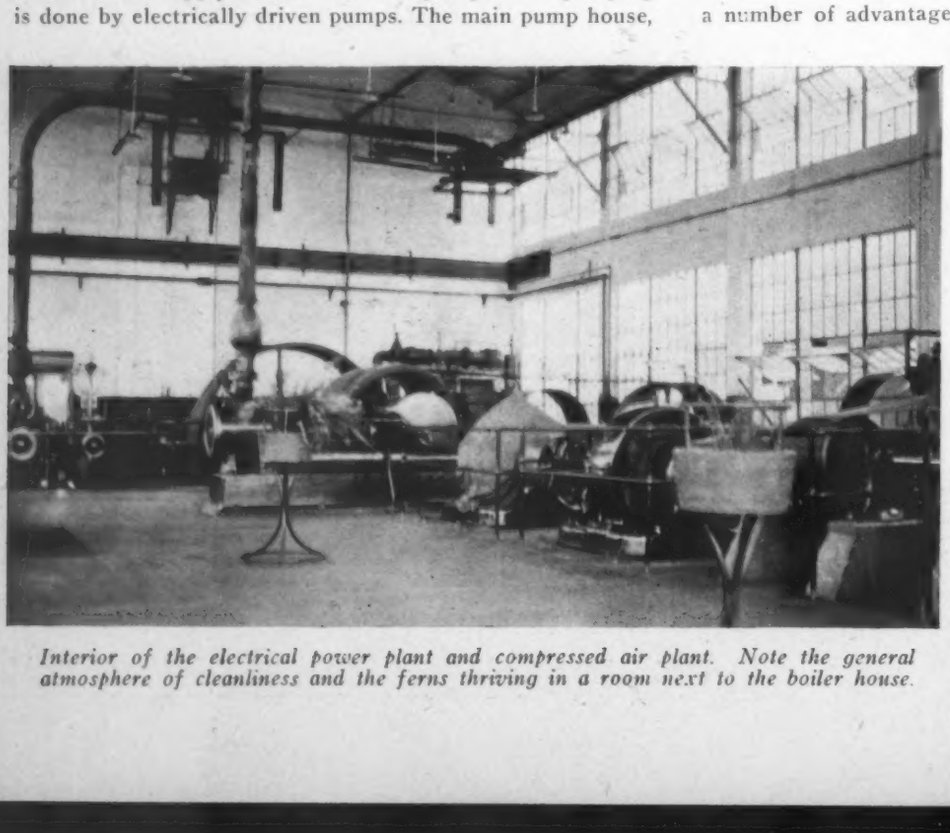


Showing details of the changes in firing the old Cross units. To the left is shown construction work on the new Gyro vapor phase cracking units.

Cracking

An interesting change in firing one of the older Cross units has been made recently at this plant. Instead of firing the unit at each end as is common practice in Cross still operation the Pure Oil engineers equipped one of the old furnaces with large Dutch ovens placed at one side of the furnace. The installation is shown in an accompanying photograph. A second small Cross still fired in the usual manner is shown beside the newly equipped furnace. At the left back ground is shown construction progress of the new Gyro unit.

With the Dutch ovens provided at the side of the furnace a number of advantages have been noticed after about ten days operation. The smoke was moved from the end of the furnace to the center, and but one arched baffle wall is placed between the preheating bank and the main cracking bank of tubes. An appreciable saving in fuel was secured through the use of the dutch ovens and side firing based on former throughput. However, since the changes have been made the throughput was easily increased 30 to 35 per cent. above the former run. Better combustion is secured in the furnace. There is no flame on the tubes, which increases the life of the tubes and decreases tube failures, and at the same time the stills can be fired heavier without injury to the tubes. The change is inexpensive and secures much better distribution of the heat. Six gas burners are provided for firing. Further, the new system is much



Interior of the electrical power plant and compressed air plant. Note the general atmosphere of cleanliness and the ferns thriving in a room next to the boiler house.

Interior of the main pump house showing battery of triplex pumps operated by electric motor driving a single shaft. Manifolds are just opposite the pumps on the other side of the house.

safer for the operators, since the fireman is not directly under the ends of the tubes at any time. In the event of fires occurring at the ends of the furnace the burners may be turned out and the firing of the unit stopped without the usual hazard involved in end firing.

In the operation of the larger and newer Cross units which have a rated capacity of 2000 barrels, the company has effected an increase in throughput by the use of several features perfected by company engineers, to the end that 3500 to 4000 barrels are passed through the units daily. Charging stock is both gas oil and fuel oil.

Quick Cleaning

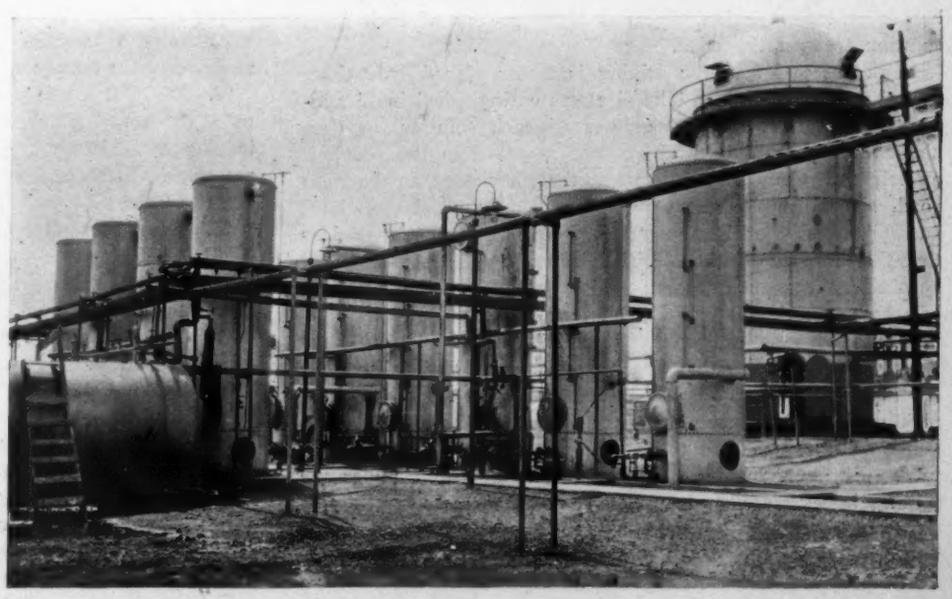
In the matter of cleaning these large units the company has reduced the down time remarkably and are limiting the down time, from stream to stream, to but ten hours. During the summer months the units are taken off stream at four in the morning and are usually fired up again at noon. Because of the high velocity of oil flow through the tubes, but little carbon is deposited at this point. In addition, it is ordinary practice to start cleaning tubes within two and one-half hours after the fires are cut. This gives hot tubes to work with, and it is thought that the relatively high temperature of the tubes has much to do with the ease with which they are cleaned of carbon. The workmen use special asbestos gloves when cleaning tubes, and use a hose insulated with asbestos.

The reaction chamber is also cleaned while hot using a system perfected by the company engineers which consists of blasting the coke from the chamber by means of high pressure water directed into and against the coke deposit.

Because workmen begin the cleaning operation so quickly after the units are shut down, and finish the job in such a short time, the system does not have time to get cold, before it is again placed in service. Usually, at the end of the cleaning time, the furnace temperature is around 500 to 600 in the fire box, with the rest of the equipment correspondingly hot. Because of this the units are on stream within two hours after starting the fires. The furnace temperature during operation is maintained between 1800 and 2000 F. In view of the fact that the company charges fuel oil as well as gas oil to these units; operates them at approximately double their rated capacity; using high temperature, pressures, and velocity of flow, and then accomplishes such remarkable results in cleaning time with a shut down period of but ten hours from stream to stream, the installation is certainly one of much interest.

Another interesting item in conjunction with the Cross unit operation consists of the long service secured in the matter of packing for the large hot-oil charging pumps. Former practice consisted of packing these large pumps at the end of every run, which is not only comparatively costly, but is a slow and tedious task. Through the installation of a series of small lines running from a common tank to all four pumps, cold oil is conducted to the pistons of the pumps at the packing gland. The small lines carry the cold oil downward through the water jacket and directly to the moving piston. Small valves are provided above each inlet, by means of which the volume of cold oil, which is very small, is controlled. Since the installation of this system of injecting cold oil to the piston at this point, the charging pumps require packing only at the end of five to seven months of practically continued service.

Gasoline from the Cross units is removed direct, and is a finished motor fuel except for doctor sweetening at the treating plant. This consists of a continuous treating system of usual design, having a series of treating and washing towers provided with the various required mixing and control devices.



Chemical treating plant assembly. The continuous treaters to the left are employed for sweetening gasoline.

Extension Tube Adds Efficiency To Foam Equipment

By BRAD MILLS
Staff Representative

THOUGH perfected more than a year ago by the Standard Oil Company of California, the asbestos extension tube still stands as one of the most efficient contributions to fire extinguishing equipment, as applied to oil tank fires. This apparatus consists of a flexible asbestos tube and is attached to the gooseneck end of the towers or portable mixers used for carrying foam over the top of a burning tank. It is the result of a series of experiments conducted by the company after a 115-foot diameter tank of burning distillate at El Segundo defied all efforts of a trained crew and burned itself out.

Some of the events leading up to the development of the asbestos extension tube follow:

Soon after the tank of distillate burst into flames, the foam system was brought into action. The fire seemed to die down after the foam was applied, with every indication that it would soon be out. The blanket of foam, after a fairly good smothering effect, seemed to lose its effectiveness. It was observed that the mixing box in the tank had been partially tipped over when the tank roof blew off, and it was immediately suspected that the two solutions were not working properly together. Portable mixing tubes were brought into play after the solutions were almost completely shut off from the regular mixing box. The foam from these portable mixing tubes also checked the fire for a time, but the blaze continued, although about three and one-half feet of foam blanketed the tank.

The company was extremely dissatisfied with the progress made against the El Segundo fire and decided to conduct an extensive investigation as to the cause of the failure. Since it was impossible to account for the failure merely from observation, the company used what remained of the tank for making what it termed "full scale tests." A small amount of oil was placed in the tank, the mixing box placed about 20 feet above the oil and tilted at about the same angle as it had assumed during the fire. The fire was extinguished after an hour's battle, but it had required 33,000 gallons of each foam solution to turn the trick.

In the second test, the mixing box was placed only three feet above the oil level. The fire was extinguished in 13 minutes with a total of 8400 gallons of each solution, or only about 25 per cent. as much as was required in the previous experiment. Company experts saw that they were on the right road, since dropping the mixing box almost to the oil level had produced amazing results. "This confirmed suspicions that the difficulty in the original fire and in the first test, had been that the foam dropping a long way onto the oil surface splashed so violently into the oil that it became oil-saturated, and thus lost most of its effectiveness."

The company knew that fires might occur in tanks where the oil level would be 10 or even 20 feet below the top of the tank, and realized that some arrangement should be perfected to gently pour the foam onto the oil, rather than let it splash as in the El Segundo fire. Clumsy baffles or floats under the mixing boxes were suggested, but it was found that the expense of equipping the many tanks with such a device would be very heavy.

Company engineers finally agreed upon some form of an extension tube which would be placed on the end of the portable mixers. It took some time to design a hose flexible enough to coil up compactly and strong enough to stand the initial rush of the solutions. The asbestos extension tube was the result of a series of experiments, during which strength, flexibility and general design were studied. Wire-inserted asbestos cloth is used. A mixing basket is placed at the bottom of the tube, allowing the solution to gently roll out over the burning oil without danger of saturation.

In the final tests with the new equipment, the fire was completely extinguished with only 3300 gallons of each solution, or about one-tenth of the amount necessary to extinguish a similar fire with the mixing box 20 feet above the oil surface, and less than half the amount required to extinguish the fire when the mixing box was only three feet above the oil. The rate of delivery of the solution was one-third of the rate that had been required in former tests.

The Company has compiled complete directions for using the asbestos tube in connection with the portable foam tower. Each specific direction is illustrated with an actual photograph, a total of 12 operations being necessary to place the asbestos extension tube and portable foam tower in action against the fire.

The directions, as issued by the company, follow:

"NOTE: Twelve trained men can handle tower, but under fire conditions at least 20 men should be used. Men should wear cotton gloves. Speed in preparing and erecting tower is not necessary, but it is essential that no time between the introduction of the asbestos to the fire and the arrival of the foam into the bowl. Bowl should be within a foot or two of surface, but *must not be in the oil.* Allowance must be made for expansion of fuel and crude oil when foam is applied.

"1. At any point near the fire where work can be done in comfort, lay tower flat on its back with gooseneck vertical and the three poles extending beyond gooseneck end of tower. Attach the three guy cables but keep these wound on spools. With knife or tinsnips cut off top of asbestos tube at a distance from the bottom of bowl *not greater than the tank outage.*

"2. Without cutting the four lengthwise cables inside the tube, cut away about 12 inches more of the asbestos cloth, leaving the cables extending about a foot beyond the end of the cloth. Remove clamp from end of gooseneck; draw a few inches of the asbestos tube over the mouth of the gooseneck.

"3. After fastening cables to lugs replace clamps.

"4. Fold asbestos tube on gooseneck, using care to cover all projections with first folds (this to prevent straps and the rim of bowl from becoming caught.)

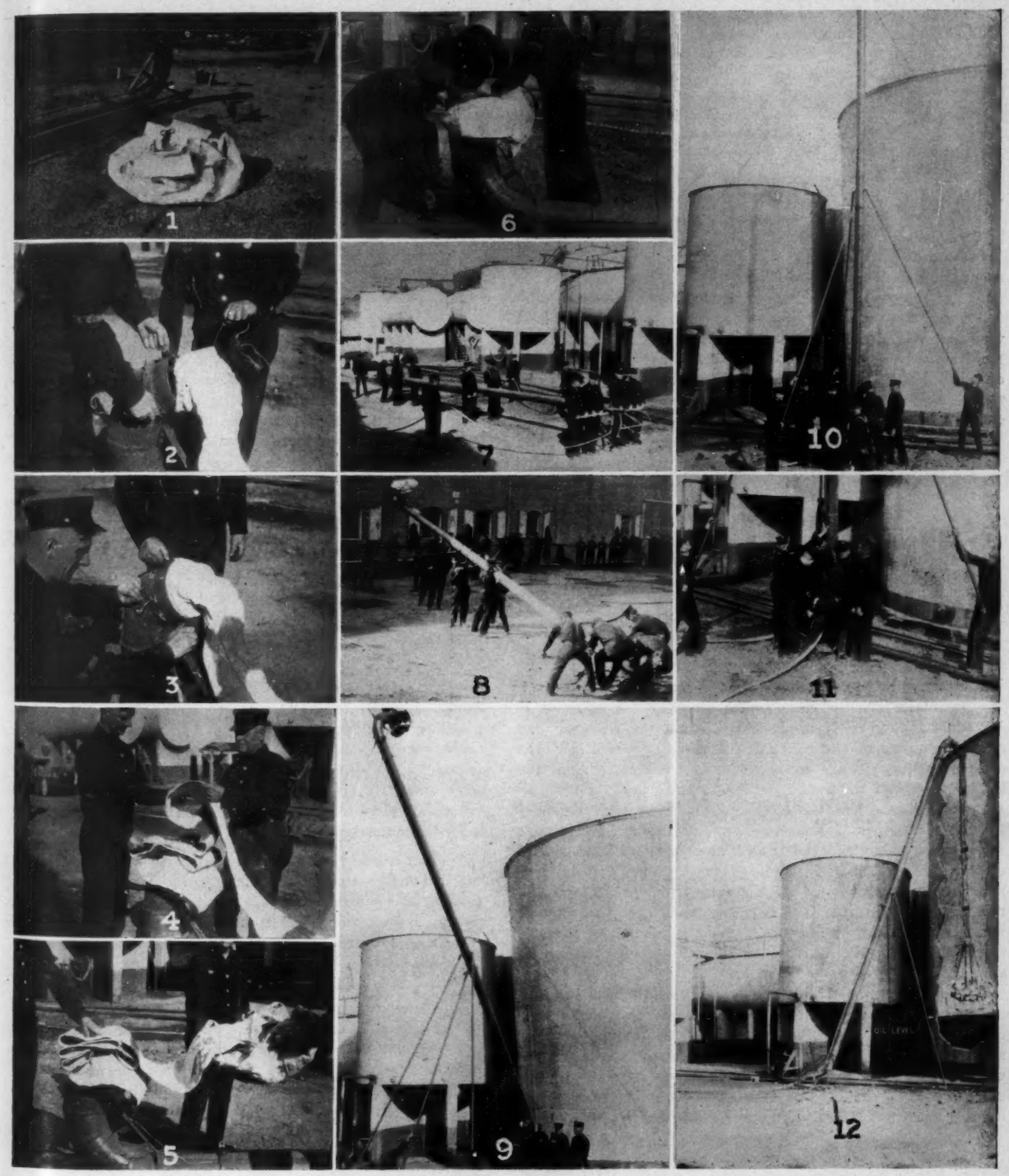
"5. Tuck in all straps and rim of bowl before placing bowl on top of folds.

"6. Hold folded tube snugly on end of gooseneck by means of cable drawn tightly over top and then attached to pin in release device. See that cotter-pin holding release plug is in such position that a pull on the cable attached to cotter-pin will allow the asbestos tube to fall free—the release cable is stretched taut and tied to the base of the tower.

"7. At the nearest set of hydrants on the foam system, install portable meters* so that solutions can be proportioned equally to the tower. Attach a 2½-inch hose line to each of the connections at the base of the tower; one hose line being

*Not necessary where total flow rate is not in excess of 250 g. p. m. of each solution.

Steps in Laying Foam on Burning Oil Without Splash



Details of these operations are given in the text.

run from the acid solution hydrant, the other from the soda solution hydrant. No more hose should be connected than is necessary to reach from the hydrant to the base of the tank where tower will be erected. The tower should be carried with base foremost and poles extending backward beyond the gooseneck. One hose line is trailed on each side; kept out of the way of the tower carriers by the hoseman. One man assigned to each of the three buy cables should carry the cable which should be extended into position as soon as the tank is reached. The tower should be laid flat on its back with the base a few feet from the shell of the tank and inspected to see that release cable is clear and that all is in readiness for erection.

"8. All of the men except the three carrying the guy cables should assist in raising the gooseneck end of the tower and steadying the base. When the gooseneck is raised too high

for the end of the men on the reach, the three poles attached to the towr should be used, two men on each pole.

"9. The center one of these poles should be kept directly underneath the tower and the two side poles a few feet on each side of the center pole. The side guy cables should be brought as far to each side as possible, these cables being left slack with only sufficient tension to prevent the tower from leaning toward the side.

"10. The center cable should remain slack until the vertical position is reached when the men assigned to this should extend the cable to its full length away from the tank and prepare to hold back the tower so that it may be lowered gently to the tank.

"11. When the tower is in the vertical position, it should be temporarily held and if necessary the base should be moved forward or back to permit the gooseneck to come in proper position on the tank rim when the tower is leaned against the tank. If the tower has not been erected from a position normal to the tank, it should be rotated to this position when held vertical. While the tower is still in the vertical position with the drain valve in the base open, the solutions should be turned on until hose lines are filled and foam flows from drain valve. Solutions should then be shut off and drain valve closed after the tower is bled empty.

"12. The tower should then be lowered gently against the shell of the tank with gooseneck snug against the tank rim, *the asbestos tube properly released, and the solutions immediately turned on.* The tube will become damaged by the heat if allowed to remain in the fire before solutions are turned on. The two solutions should be proportioned equally, using about 200 g. p. m. of each solution if this rate is available.

NOTE: Men should be assigned to positions as follows:

1 to each solution hydrant	2
2 to each side guy cable	4
3 to center guy cable	3
2 to each pole	6
4 to base of tower (2 of these will operate valves and one the release device)	4
Total	19

"Ten of these men will carry tower and three will carry guy cables. The remainder can help carry the hose, which requires about one man for each 25 feet of each hose line."

The Standard Oil Company's policy of allowing other oil companies to use their fire extinguishing processes free of charge applies to the asbestos extension tube. A complete set of drawings and permission to build the equipment described in this article will be furnished upon application to H. H Hall, Chief Engineer, San Francisco.

Separating and Concentrating Acid-Sludge

By H. L. KAUFFMAN

IN line with the progress being made in the operation of all refineries are the developments that are being made in the handling of by-product materials. Acid-sludge is one of those substances, the economical disposal of which has been causing the progressive refiner no small concern. Until the last few years, separation of the acid from acid-sludges was looked upon a being advantage merely because it made possible the subsequent storage in dumps of the acid-free sludge (this sludge was later burned) with no complaints from owners of surrounding land. Now, however, with modern developments in methods of separating acid from acid-sludge and thereafter concentrating the separated acid, and with suitable means devised for burning the remaining sludge as fuel, refiners are finding the recovery of such acid to be an economical venture.

However, there are still a few refiners who do not have installed in their plants the most recently-developed types of acid-concentrating equipment. To familiarize operators, engineers and executives of such companies both with the various means of separating acid from acid-sludge now in use and with the different kinds of acid-concentrators devised in recent years is the primary purpose of the present discussion.

Handling of Acid-Sludge

The amount of sludge that results from the treatment of lubricating oil with sulfuric acid will vary from as low as 3 per cent. in the case of sludge from the treatment of non-viscous lubricating distillates to as high as 35 to 40 per cent. in the treatment of cylinder stocks. Disposal of this sludge is one of the most serious problems with which the refiner must deal. Dumping this material into a pond or attempting to discharge same through the usual outlets for sewage soon creates an intolerable nuisance. As previously mentioned, in the past, large quantities of sludge have been burned in open ponds with no attempt to utilize the calorific value of the sludge. More recently, specially-designed furnaces have been employed for handling the sludge as fuel. So, too, improvements both in burner design and in technique of firing furnaces have made it possible to burn certain classes of sludge under boilers and stills with fair efficiencies. However, the nuisance resulting from soot and fumes makes such practice unpopular in thickly-settled communities.

Modern refinery practice consists in separating the acid from the acid-sludge, concentrating the recovered acid for re-use and utilizing the neutralized sludge, most often as fuel. However, paving asphalt and other special products can also be made from this sludge.

Composition of Acid-Sludge

The composition of acid-sludge is very complex and it is difficult even to conjecture of what it consists. It is known, however, that this sludge varies in its composition and characteristics depending upon the nature of the oil being treated, the method of treatment, and the quantity and strength of the acid used in the treating process. Briefly, though, acid-sludge may be classified as being an emulsion or solution of tarry matter in unspent acid which, in turn, holds in solution sulfuric-addition products.

Careful investigation work has shown that acid-sludge mechanically absorbs some non-reactive paraffines and naphthenes. These hydrocarbons under ordinary conditions do not react chemically with sulfuric acid of any strength. Likewise, we find in the sludge appreciable quantities of sulfonic acids and alkyl-sulfuric acids resulting from the action of sulfuric acid on the aromatic hydrocarbons in the oil. We also find present various organic acids, such as naphthenic and poly-naphthenic acids, which sulfuric acid seems to remove more or less me-

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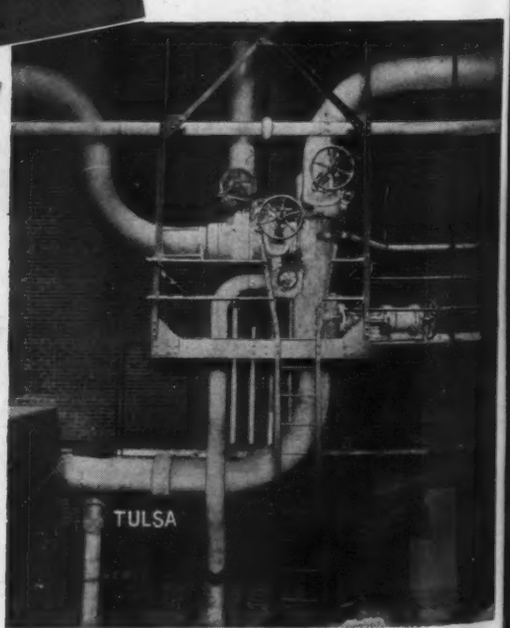

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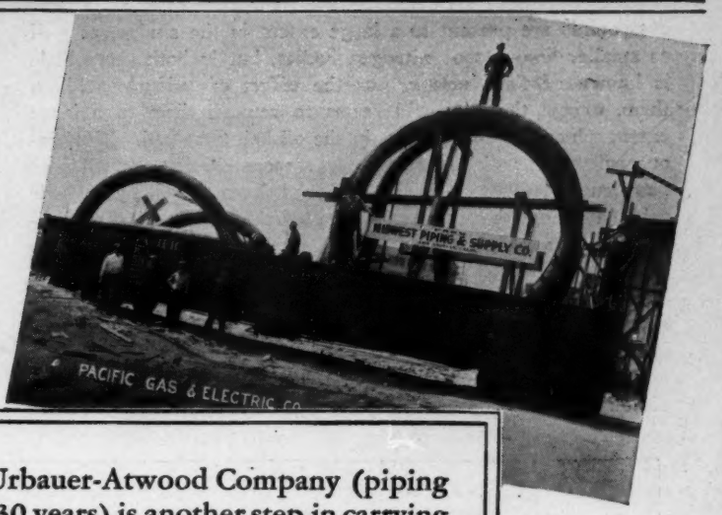

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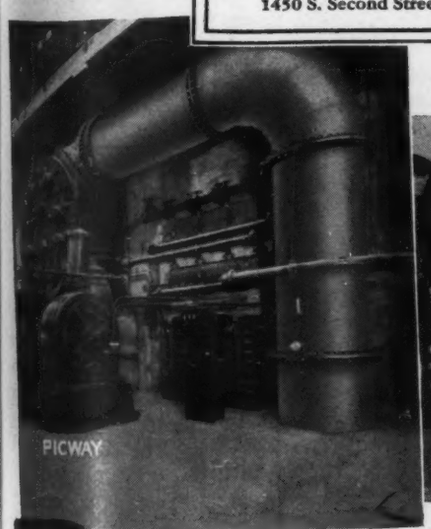

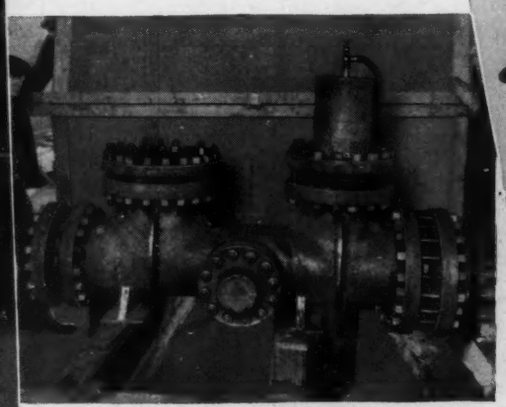

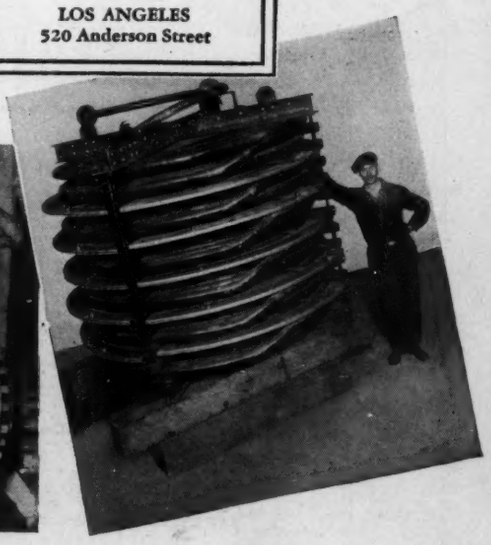

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chanically without greatly affecting their composition. Sulfur compounds are present to a large extent in the sludge, as well as smaller amounts of nitrogen bodies, but in both cases little is known of their nature, nor the effect of sulfuric acid on them, except that the acid seems to remove them to a large extent when they are present in the oil being treated. Bituminous substances, which are largely responsible for the color in petroleum distillates, are also removed from the oil by sulfuric acid (although the exact chemistry of the reaction is not definitely known), and they, too, are to be found in the acid-sludge. The chemical reaction between sulfuric acid and the unsaturated hydrocarbons comprises polymerization to dipolymers and the formation of esters and hydrated esters of sulfuric acid, the latter on treatment with water forming secondary and tertiary alcohols. All of these compounds may be found in the sludge, although, according to Brooks and Humphrey, the dipolymers are more likely to find their way into the oil layer, for they are only slightly dissolved by sulfuric acid.

Separation of Acid From Acid-Sludge

The recovery of free sulfuric acid from sludge discharged from the agitators, which sludge is a dark, viscous, tar-like liquid, necessities separating the acid from the oil and tarry products. The sulfonic acids contained therein are usually decomposed by diluting the sludge with an equal volume of water, heating the mixture to about 180 degrees F. and then agitating the whole with high-pressure steam and air until no more sulfur dioxide is given off. By this cooking process, most of the sulfonic acids are broken up. The resulting acid can then be concentrated to any desired strength up to 66 degrees Baume without further treatment.

The equipment required for sludge-acid separation naturally depends on the character and quantity of material to be handled. The sludge is diluted and cooked in chemical, lead-lined vessels having open steam-coils and draw-off connections for oil and acid. The tanks may be equipped with either cone or slanting bottom to suit local conditions, but they should be placed in an elevated position to permit the decanted material to flow by gravity into the fluxing tank and acid run-down tanks. Ample storage should be provided for the weak acid, since only well-settled and skimmed acid should be fed to the concentrator.

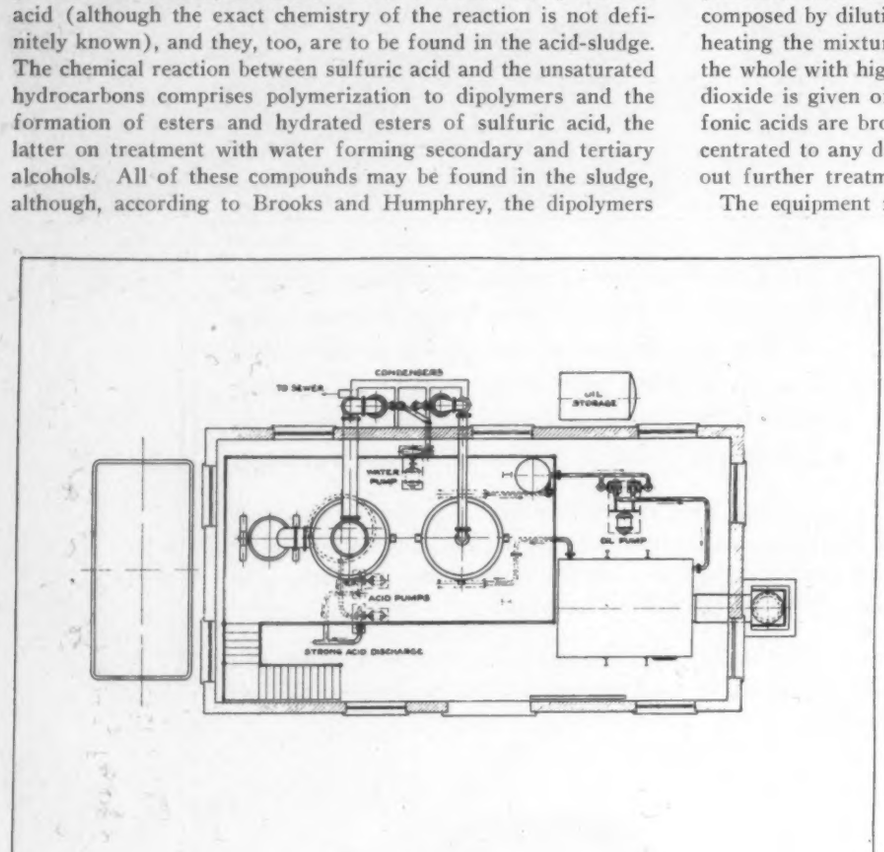

Figure 1.
Simonson-Mantius sludge acid concentrator.

The size of the separator used depends upon the size of the agitator that it serves, but generally varies from 100 to 300 barrels in capacity. A separator of the latter size would serve satisfactorily a 1000-barrel agitator. It is advisable to have at least one-third excess capacity in the separator. The separators are lead-lined and are equipped for agitation either with air or live steam. Separators 15 feet deep and 15 feet in diameter are of a size frequently used by refiners. The contents of the separators are heated either by the direct introduction of live steam, or by closed steam-coils placed within the kettle proper, or by both. All steam and air-lines are made of lead. Because of the fact that complete separation of the acid may require as much as two days' time, it is usually advisable to employ two separating tanks for use with an agitator in which one batch of oil per day is treated; and four tanks, if two batches are treated in the agitator each day. The separators are equipped either with a lead plug, about four inches in diameter, which fits into the "draw-off" at the bottom, or with a valve made of some acid-resisting material. In the former case, the plug is raised to discharge the separator by pulling down on a chain that is attached to a lever which, in turn, is fastened to the top of the plug-rod. The viscous tar-like liquid that settles-out of the treated oil is either pumped from the agitator to the separator or is blown-out with live steam. The draw-off pipe may or may not be steam-jacketed. The disadvantage of not having a steam-jacketed line lies in the fact that when live steam is used in blowing the sludge from the agitator to the separator,

there is a tendency for the condensed water to dilute the acid and a subsequent inclination of the acid to attack the pipe more readily.

The practice followed by one refiner in the treatment of the sludge in the separators is as follows:

The sludge when first added to the separator is heated both by means of a closed steam-coil and by blowing live steam directly into the sludge until same is liquefied. This condition can be determined by punching around in the separator with a long stick. The contents are then allowed to settle for about one or two hours at a temperature ranging from 150 degrees to 200 degrees F., after which the greater portion of the acid settles and may be drawn-off through a three or four-inch lead-lined pipe that is welded to the bottom of the separator near the top of the plug. The sludge is next washed with water to remove more acid. These washings, thereafter, are passed to the acid vats. Washing of the sludge is continued until the average gravity of the acid in the vats is such that further dilution would not be economical. Washing is never continued to such a point that the separated acid is lower in gravity than 30 degrees Baume (for liquids heavier than water); and, as a matter of fact, the gravity usually ranges from 33 degrees to 35 degrees Baume.

The separated weak acid obtained in the above-described manner is very dark in color and contains certain organic compounds resulting from the treatment of the oil. In this condition it contains only about 36 per cent. of concentrated sulfuric acid, water being present in the greater proportion. The amount of sulfuric acid recovered depends entirely on the nature of the sludge and the quantity of sulfonic acids contained therein. The results will vary with the grade of oil treated and the quantity and the density of the original acid used for treatment. Light-oil sludge has been known to yield as high as 80 per cent. of the total acid originally present in the sludge; lubricating oil sludge, about 65 per cent.

In the experience of another refiner[1], efficiency recovery of acid from sludge oil demands a process whereby it will be possible to heat sludge to temperatures sufficiently high to effect rapid hydrolysis (splitting-up of sulfuric-acid addition products by boiling with water to form sulfuric acid and neutral organic compounds) without permitting the escape of water vapor and the consequent destruction of acid. This can be accomplished by heating under a pressure slightly higher than the pressure exerted by steam over acid of the strength being recovered. The exact temperature, proper reaction time and dilution of the acid to be produced, in this method of hydrolization, will depend upon the type and the properties of the sludges being treated. Thus, some naphtha sludges require only one hour's treatment at 275 degrees F., producing 40 degree Baume acid; cracked-naphtha sludges, one hour at 320 degrees F., producing 45 degree Baume acid; while a sludge produced in the treatment of kerosene with fuming acid at 150 degrees F., requires two hours' time at 340 degrees F. for hydrolysis, producing 49 to 50 degree Baume acid. The pressures at which these operations are carried out vary from about 25 pounds for the naphtha sludge to as high as 65 pounds for the fuming-acid kerosene sludge. Operation of the plant is continuous.

The retort used in this method of separating acid from acid-sludge has a capacity of 100 to 125 tons of 40 degree Baume acid. This retort consists of a steel shell, approximately 30 feet long by six feet in diameter, lined with heavy sheet-lead which, in turn, is overlaid with acid-proof brick set in acid-resisting cement. Such construction insulates the lead and protects it against corrosion and mechanical failure. Sludge, water and steam are intimately brought together in a mixing device, and are then passed into the retort or digester. The acid and tar separate into layers in the retort and may be withdrawn independently. By suitable arrangement of the acid and tar outlets, any desired ratio of acid-volume to tar-volume may be maintained in the retort. The ratio of these same constituents as they enter the digester is fixed by the composition of the sludge being retorted. If a mixture that results in the production of equal volumes of weak acid and tar is being charged in any batch process, the tar and acid both will be subjected to the same time-temperature treatment. On the other hand if, in the same case, the tar is withdrawn almost as fast as it separates (thus keeping a thin layer in the retort), the mixture will pass through the apparatus at a high rate. This is due to the fact that the mixture is subjected to the effect of the high retort temperature for a relatively short time only. The tar, therefore, will be less subject to oxidation: consequently, it leaves the retort in a more fluid condition than otherwise would be possible. Another benefit derived is that the acid separates from a limpid tar more rapidly than from a viscous, stringy tar. By keeping the volume of tar in the retort reduced, the volume of acid which it is possible to carry in the apparatus is nearly doubled; as a result, the average throughput time of the acid is increased. This gives the mixture a relatively long digestion at high temperatures, a condition that is essential to the production of clean acid. Pressure retorting of sludge, in the experience of this refiner, almost doubled the yields of weak acid as compared with those formerly obtained. A further advantage claimed for the process is that on account of the low carbonaceous content of the acid, concentrating difficulties have been largely overcome.

[1]From a paper, "Recovery of Acid from Sludge Oil," read by W. N. Davis of the Standard Oil Company of California, at the December, 1927, meeting of the American Petroleum Institute at Chicago.

Methods of Handling Separated Acids

In most refineries the weak acid coming from the separators at a density of about 30 degrees Baume is first concentrated either to 56 degrees or 61 degrees Baume. This partially-concentrated acid is then allowed to settle, so as to remove the tarry matter which has been separated by the decomposition of sulfonic acids during the first stage. The settled acid is then further concentrated in a second apparatus to a finished product of 66 degrees Baume.

Many refineries use both 60 degree and 66 degree Baume for treating oils. In such cases, it is more economical to concentrate to 60 degrees or 61 degrees Baume only, use a part of this acid for the treatment of light oils, and reinforce the balance to 66 degrees Baume by blending with fuming sulfuric acid (containing 20 per cent free sulfur trioxide.)

Where light oils and cracked distillates are treated with 60 degree acid only, it is possible to recover the acid in a simple and inexpensive apparatus. Even small refineries handling a few tons of acid per day can recover this acid economically. Refineries operating a contact sulfuric-acid plant for the production of their make-up acid can recover the separated acid by concentrating to 61 degrees Baume, and then using this acid in the absorbers of the acid plant where said weaker acid will be reinforced by sulfuric trioxide either to 66 degrees Baume, 98 per cent. acid or 20 per cent. oleum (fuming sulfuric acid), as may be desired.

Concentrating Recovered Acid

The pan-and-still system of sludge-acid concentration has been supplanted largely by other methods. This has been due to the fact that, in comparison with other processes, in the pan-and-still system the fuel efficiency is low; the labor costs are high; maintenance costs are excessive; the percentage of recovery of the acid is low (usually about 80 per cent., occasionally as high as 85 per cent.); and, finally, obnoxious sulfur trioxide fumes are given-off.

The Simonson-Mantius Vacuum Process consists in removing the water of dilution and concentrating the weak sulfuric acid to the desired density in a closed vessel under a relatively high vacuum by the application of indirect heat—high pressure steam or hot oil most commonly being used as the source of heat. The use of vacuum is especially applicable to the concentration of sulfuric acid. This is due to the fact that distillation under vacuum reduces the boiling point of the acid to such a level that heating mediums of comparatively low pressure and temperature can be used. This reduces the cor-

rosive action of the acid and greatly simplifies the construction of the apparatus. The difference in boiling-points of sulfuric acid at various densities and pressures is illustrated by the fact that at a density of 30 degrees Baume, the boiling-point of the acid at 28 inches vacuum is 120 degrees F., as compared with 230 degrees F. at atmospheric pressure—a reduction of 110 degrees F. Under similar conditions, the reduction in boiling-point for 61 degree acid is 150 degrees F.; for 66 degree acid, 180 degrees F. These low temperatures make it possible to concentrate sulfuric acid to 61 degrees Baume with steam at 45 pounds pressure; and to 66 degree Baume, with hot oil at a temperature of 500 degrees F. There is no difficulty in constructing extra heavy, lead coils to operate with a steam pressure of 45 pounds; while any high grade lubricating oil with a flash-point of 550 degrees F. is suitable for the final concentration of the acid. This reduction in temperature reduces the wear on the apparatus, in view of the fact that chemical lead of good quality is not corroded by 61 degree Baume sulfuric acid when the temperature does not exceed 300 degrees F.

In the Simonson-Mantius process the vacuum is created and maintained by special, barometric-type jet condensers: no vacuum pump is required. In this process, too, the acid is usually concentrated in two steps. This is done in order to permit settling of the acid at a density of about 60 degrees Baume. The weak acid is drawn by vacuum into the preliminary concentrator, which is heated by steam at from 30 to 45 pounds pressure. As soon as a density of 56 degrees to 61 degrees Baume has been reached, the vacuum of this unit is broken and the acid is discharged by gravity into a storage tank. The settled acid is again drawn by vacuum into the final concentrator, which is heated by hot oil circulated through an oil-furnace of standard design. When the acid has reached a density of 66 degrees Baume, the vacuum of this unit is broken and the acid discharged by gravity into the cooler and run-down tank. Where large quantities of acid are to be handled, it is possible to operate the concentrator continuously. Figure 1 shows the design of a Simonson-Mantius sludge-acid concentrator and auxiliary equipment.

Various types (four in all) of concentrators are recommended for the concentration of weak acid to different degrees of strength. Each type, however, is especially designed and constructed for the purpose for which it is to be used. In apparatus for the concentration of either 56 degree or 61 degree acid to 66 degrees Baume, hot oil is employed as the heating medium. By using high-pressure steam of 100 pounds inside the tubes, 63 degree Baume acid can be produced from weak or partially concentrated acid. The construction of the shell is of standard design. The heating surface consists of special, high-silicon iron tubes, placed vertically in the shell, and bolted to flue-plates by means of hollow-tube fittings. These fittings, in turn, serve as inlet and outlet for the heating medium. The flue-plate of the concentrator is made of special, acid-resisting cast-iron, the top surface of which is further protected by several layers of acid-proof tile laid in Acitite cement. The tube openings proper are lined with high-silicon, iron sleeves also set in Acitite cement. This concentrator can be operated either in batches or continuously, depending on the quality of acid to be handled.

The claimed advantages for the Simonson-Mantius vacuum process of concentrating sulfuric acid are as follows:

1. *Absence of fumes and bad odors. The low boiling temperatures prevailing in the system almost entirely prevent the decomposition of acid. Further, no fumes can escape into the atmosphere, in view of the fact that any sulfur dioxide and sulfur trioxide fumes given-off during the process of concentration are absorbed in the cooling water in the condensers.*

2. *No acid leaks. Because of the vacuum maintained in the whole system, air leaks are impossible. Should any joint become defective, air will be drawn-in and the vacuum, gradually, will go-down. This gives the operator ample time to discharge the acid into storage tanks. Therefore, foundations and buildings can not be damaged by acid escaping from the system.*

3. *Low first cost and depreciation. The equipment used in handling sulfuric acid up to 66 degrees Baume at boiling temperatures is usually very expensive, in view of the fact that in order to withstand corrosion all parts throughout must be made of exceedingly high grade material. However, it is claimed that because of compactness of equipment the Simonson-Mantius concentrator can be installed at reasonable cost. Depreciation charges, therefore, do not influence materially the cost of acid recovered. An allowance of 20 per cent. for depreciation and interest is said to be ample.*

4. *Small floor space required. The apparatus requires only a small floor space: about 1000 square feet, for a 20-ton plant; and about 1500 square feet, for a 50-ton unit. The clearance between roof and ground floor is about 36 feet. Where climatic conditions permit, no housing is necessary for the concentrator. In any case, a plain building of structural steel with corrugated steel roof and siding is entirely satisfactory. Such a building can be erected at low cost.*

5. *Can be located conveniently. Because of the complete absence of all fumes, the concentrator can be placed where it is most convenient, that is, near the agitators or separating plant.*

6. *Low operating cost. Since the acid is concentrated in a closed apparatus at reduced temperatures, the fuel consumption is quite low and the radiation losses are small. Depending upon the density of the weak acid, the total fuel consumption varies from 50 to 70 gallons of fuel oil per ton of 66 degree acid produced. This amount can be reduced materially where exhaust steam is available for the preliminary concentration. Depending upon the water temperature, the cooling-water consumption will vary from 20,000 to 30,000 gallons per ton of 66 degree acid. On account of its low acidity, the cooling water can be re-used, if necessary. In such cases, however, about 10 per cent "make-up" water should be added daily. The total power consumption is from 20 to 25 KW hours per ton of 66 degree acid. This includes the power required for pumping the cooling water. Repair costs are kept moderate by the careful selection of suitable material for the construction of the apparatus. The circulating oil is usually replaced about once or twice a year. Consequently, it is claimed that, varying with the size of the plant, the total maintenance charges do not exceed from $0.50 to $1.00 per ton of 66 degree acid produced. When making 61 degree acid, the cost of repairs will range from $0.10 to $0.30 per ton of finished acid. As to labor costs, operation of the plant is simple and, therefore, one man per shift can operate all the equipment required for maying 66 degree acid. Concentrators producing 61 degree acid require only part of the time of one man per shift.*

7. *High efficiency. Vacuum evaporation is said to increase greatly operating efficiency, in view of the fact that the boiling-points are lowered and radiation losses are reduced. On account of the low temperatures employed in the Simonson-Mantius process, very little acid is distilled over and, therefore, it is claimed that the total efficiency of the process is from 95 to 97 per cent. This refers only to the loss of sulfuric acid. There are no means of limiting the loss of sulfur dioxide, which loss is due to the decomposition of the sulfonic acids. The amount of this loss depends entirely upon the quality of the separated acid. Simonson-Mantius plants can be started in about one hour and can be shut-down in about 20 minutes. This feature makes it possible to operate the concentrator intermittently, without appreciable losses and delay.*

Plants of this type are installed in refineries of Empire Refineries, Gulf Refining Company, Houston Oil Company, Indian Refining Company, Lincoln Oil & Refining Company, Marland Refining Company, Pure Oil Company, Standard Oil Company of Indiana, Sinclair Refining Company, Vacuum Oil Company, and Argentine Government at Buenos Ayres.

The process of sludge-acid concentration developed by the

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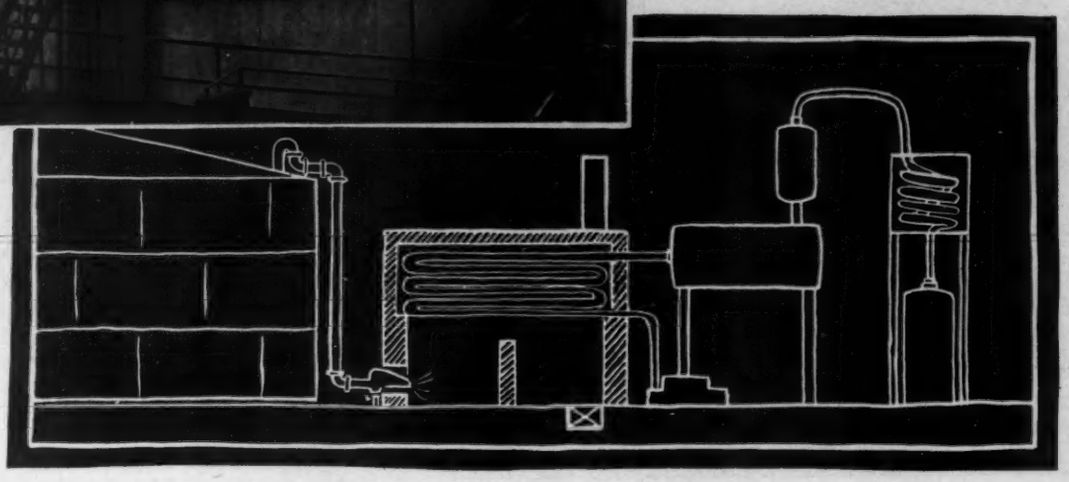

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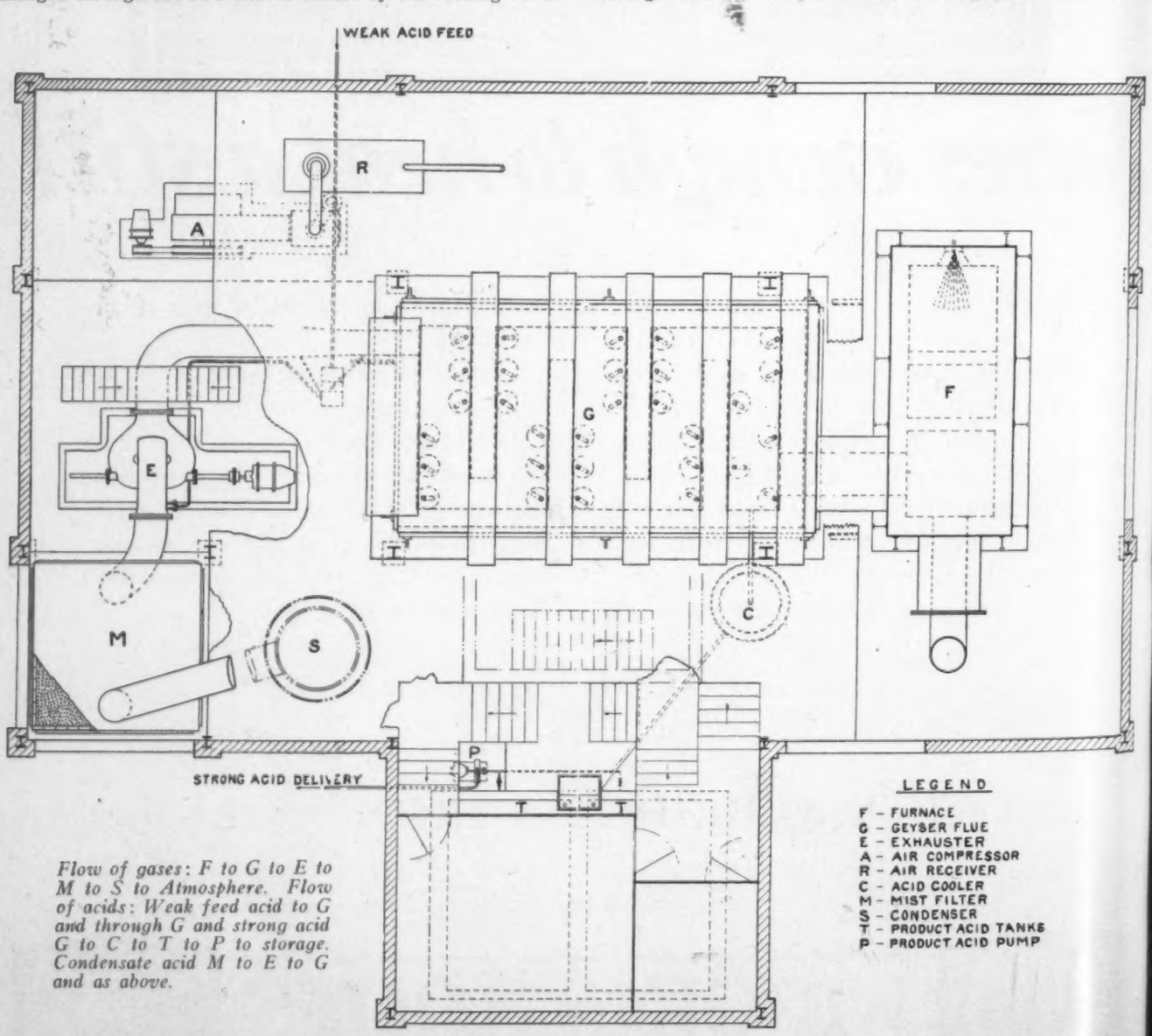
Chemical Construction Company is based upon the physical law that the boiling-point of an acid of a given specific gravity is lowered when air is introduced directly into it. In the "Chemico" process, air at a temperature of 1200 degres F. is blown through the acid-bath, where said air quickly gives-up most of its heat and emerges at a temperature only a little above that of the acid itself. Thereafter, almost all of the heat remaining in the air is usefully employed in the preheating tower. The process is continuous, except that it is necessary to divide it into two stages in order to avoid the frothing and foaming due to decomposition of the hydrocarbons present. The two-stage concentration also makes it possible to remove most of the impurities in the first stage—partly by volatilization and partly by oxidation—so that a comparatively-clean acid remains for concentration in the second stage.

Each "Chemico" type of acid-concentration plant includes two units, one for each stage. These units are almost identical in construction and are combined in one self-contained structure requiring a minimum of floor space. The sludge-acid is contained in a rectangular lead pan, lined with acid-proof brick and carried on a raised concrete foundation. About two-thirds of the area of the pan is covered by an arch of acid-proof brick, through which enter the acid-proof iron pipes discharging the hot air beneath the surface of the bath. The air discharged through the acid bath is heated by the burning of oil in a specially-designed combustion furnace operating under pressure. A blower supplies the air under pressure—not only in the quantities required for the combustion process, but also a needed excess for regulating properly the temperature of the mass and for furnishing necessary oxygen to prevent dissociation. The hot air from the furnace passes through a header to the concentrator units, where the air is distributed through manifolds beneath the surface of the acid bath. In the rear of each pan is a preheating tower of acid-proof masonry packed with brick and furnishing the surface area necessary for the maximum absorption of heat from the escaping gases by the in-coming acid. The usual acid air-lifts are used for pumping and regulating the acid supply, which enters at the top of the towers and flows downward over and through the packing rings. The gases leaving the preheating towers are passed through Cottrell electrical precipitators to recover the acid present and to prevent fume nuisance.

In the operation of this process the rate of supply of acid to be heated is regulated to control the speed of concentration and the strength and temperature of the acid bath. In the first-stage operation, by regulating the temperature and quantity of air blown through the bath, the acid strength in the pan is maintained at from 50 degrees to 55 degrees Baume, or just below the point at which foaming would occur. At this strength of acid, the hydrocarbons are largely broken-down and



Flow of gases: F to G to E to M to S to Atmosphere. Flow of acids: Weak feed acid to G and through G and strong acid G to C to T to P to storage. Condensate acid M to E to G and as above.

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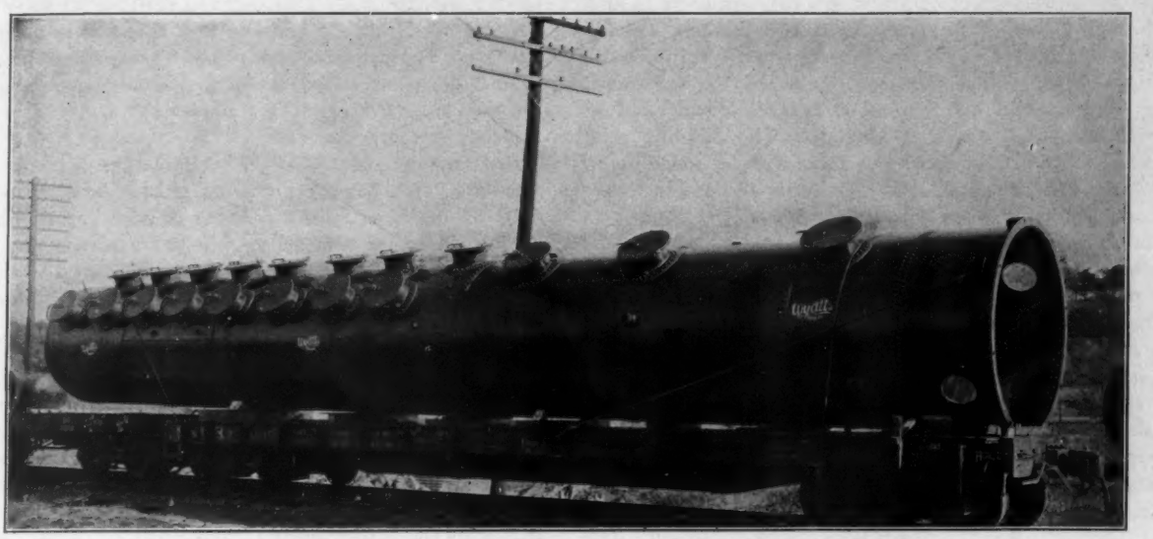

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volatilized by the rapid flow of hot air. The acid remains constantly at a strength sufficiently low so that the contained carbonaceous materials—tar, oil, etc.—are not attacked. The acid from the first-stage operation drains-off continuously and passes through a cooler into intermediate storage tanks, whence it is pumped to the top of the second-stage unit for final concentration. The preheated acid from the second or high-stage tower is maintained in the pan at 66 degrees Baume and is drawn-off continuously at that strength. The finished acid leaves the concentrator at about 110 degrees to 125 degrees F. below its true boiling-point. It is then ready for use in treating oils and distillates in the refinery processes.

Fuel oil consumption in the "Chemico" process is about 35 to 40 gallons per ton of 66 degrees Baume acid produced, when starting with 35 degrees Baume acid at room temperature. This is equivalent to about 9½ to 10 gallons of oil per ton of 66 degrees Baume acid, when starting with acid of 60 degrees Baume strength. One man per shift is all that is needed to operate even the largest plant of this type. The maintenance cost is about $0.30 per ton of 66 degree acid recovered. Costs of recovering one ton of 66 degree acid from 35 degrees Baume sludge-acid by this process are said to be as follows:

Forty gallons of oil at $0.04 per gallon	$1.60
Labor	0.20
General expense and repairs	1.10
Losses—6 per cent.—based on $8.00 per ton of 60° acid	0.48
Process	1.00
Total operating cost per ton of 66° acid produced	$.4.38

Among the refiners who have plants of this type installed are Imperial Oil Company, Standard Oil Company of New Jersey, Standard Oil Company of New York, Standard Oil Company of Louisiana, Standard Oil Company of California, Mid-Continent Petroleum Corporation, Humble Oil & Refining Company, Solar Refining Company, Atlantic Refining Company, and Shell Company of California.

"Geyser-Action" Process

For a more ready understanding of the "Geyser-Action" process of sludge-acid concentration, refer to the apparatus plan shown in Figure 2. In this process hot gases from a suitable furnace pass through a "Geyser" flue (containing a pool of the acid, which is being successively geysered-up through the gas space above the pool), and act directly upon the acid to which they are exposed, that is, upon the surface of the pool, the drops composing the geysers and the film of acid on the ceiling and walls, which latter are thrown there by the geysers.

The "Geyser-Action" fountain is produced by discharging a stream of gas upwardly through a minute orifice in a nozzle, which is submerged slightly beneath the pool level—the gas pressure and the submergence being adjusted to cause the geyser to rise to the height desired. Since the flow of the acid is counter-current to that of the gases, the hottest gases meet the strongest acid; while the cold acid entering the other end of the flue meets the coolest gases. Evaporation of the water takes place, as a result of which the liquid body is reduced and concentrated.

The gases entering the flue are added to by the large amount of water vapor and the small amount of acid vapor, while, at the same time, they are cooled greatly by the transfer of much of their heat to the liquid. As the gases which are now growing cooler and cooler approach the cold end of the geyser flue, they reach a point where the acid is no stronger than when it entered. However, the acid at this stage is at its boiling-point, having been preheated by the gases. In this preheating zone, the continuing loss of heat by the gases reduces their temperature to a point below the boiling-point of the acid vapors contained in them, thereby causing condensation of most of the acid vapors from vapor to liquid phase. This liquid is in the form of finely-divided mist. The gases then consist of the furnace gases, together with water vapor and acid mist.

These combined gases are now passed through a filter containing obstructions. This baffling material so sub-divides the gas passages that the gases flow through in many minute streams. Those minute gas streams are continually impigning against the obstructions, thus filtering the gases by contacting the mist out of them. This condensate acid ("drip" acid) is returned to the geyser flue.

Although almost all of the acid vapors are thus removed from the gases, however, in order to provide against the possibility of any objectionable quantity of such vapors being finally discharged to the atmosphere, the gases leaving the filter are passed through a condenser. This condenser is simply a gas duct of enlarged area (in which the gas travel is slow), where successive sprays of water are thrown against the gases in their passage through the duct. In reality, these sprays are "Atomizing" sprays which deliver only a small quantity of water, but in such finely-divided form that it is immediately absorbed into the gases. Such an arrangement causes the condensation and elimination of virtually all of the acid content of the gases before their final discharge to the atmosphere.

No provision for the removal of sulfur trioxide gas from the gases is needed. This is because no sulfur trioxide is formed, that is, no acid is actually split-up. Splitting of the acid is avoided by not admitting the furnace gases to the strongest acid at too high a temperature. Also, by maintaining such an active flow of acid exposed to the gases (that is, at the pool surface, on the ceiling and walls, and in the geysered drops themselves) that no portion of the acid in contact with the gases has its strength built-up to the point at which sulfur trioxide would be split-off.

In addition to the geyser flue, filter and condenser, already mentioned as being prime apparatus-elements of this process, the following auxiliary equipment is also required:

1. *A furnace, to furnish the hot bases. This is located immediately ahead of the geyser flue.*
2. *An exhauster, to supply draft. This is located between the geyser flue and the filter.*
3. *A cooler, to lower the temperature of the product acid. This is located between the hot end of the geyser flue and the product tanks.*
4. *Product tanks, to receive the cooled product acid.*
5. *A product pump, to receive the product acid from the product tanks and to deliver it to storage for re-use.*
6. *An air compressor, receiver, pressure-regulating valve and distributing lines to the nozzle pipes, which latter extend from above the ceiling of the geyser flue through it and down into the pool.*
7. *Instruments for determining gas temperatures, air pressure and flow, and for the manual or automatic control of the burner, exhauster, air compressor, geysers, etc.*
8. *A suitable supply of water (for cooling and for condensing) and of electric current are also required.*

Several plants of this type are installed (Sun Oil Company, for one) in refineries, but location of all of such plants is not known to the writer.

Handling of Acid-Free Sludge

After the acid has been separated from the sludge, the usual procedure thereafter consists in neutralizing the remaining viscous sludge with either caustic-soda or soda-ash solution, and then "mixing-off" the neutralized sludge with fuel oil. This mixture is suitable for use as refinery fuel. In some refineries the sludge is never allowed to become cold after being drawn from the agitators, in which case it is burned without admixture with fuel oil. Almost all sludges except those from cylinder stocks can be neutralized and mixed with fuel oil without any difficulty whatsoever. It is possible to remove a considerable amount of acid, remaining in the sludge after the

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separation of the major portion of the acid, by heating with live steam and blowing with air at the same time. Neutralization is accomplished by adding a solution of caustic soda or soda ash through a small line to the separator, continuing the heating and the agitation. The use of soda ash is more economical than caustic soda, but there is greater danger that the contents of the kettle will boil over. Care must be taken even with the use of caustic soda that the sludge is not too hot before adding the caustic; otherwise, the sludge may foam over the top of the kettle. As much as 10 barrels of 12 to 14 degrees Baume caustic-soda solution may be required to neutralize 100 barrels of acid sludge resulting from the treatment of pale or red oils. The contents of the kettle are then allowed to stand until the solution of caustic soda or soda ash has settled, this solution thereafter usually being discarded. The neutralized sludge is then either pumped to a tank where it may be mixed-off with fuel oil or it is mixed with the fuel oil in the separator. A 50-50 mix is the ratio usually maintained between the sludge and fuel oil.

Little difficulty is encountered in handling sludges resulting from the treatment of pale and red oils, but considerable difficulty is often met with in disposing of cylinder-stock sludges. This is because of the nature of the sludge itself, in view of the fact that it is a very heavy viscous material when hot, and almost coke-like in its appearance when cold. In this case, the ordinary equipment usually available for disposing of it is not satisfactory, and a slightly different procedure must be followed.

In refineries where this heavy cylinder-stock sludge is not mixed-off with fuel oil, its disposal becomes a very serious and costly operation. At some refineries mono-rail cars tranfer the sludge to the boiler-house where it is burned merely as a method of disposal and with no thought of any fuel efficiency. The acid remaining in the sludge attacks everything with which it comes in contact, and for this reason the cost of repairs is exceedingly high. This procedure is only followed where no land is available for storing the sludge.

Other refiners haul the sludge from the agitators to a suitable dumping ground where the sludge is piled up year after year. Such a procedure frequently brings complaints from those surrounding farmers, whose crops are injured by the rains washing the acid from the sludge onto their soil.

Some refiners take advantage of the fact that, if the sludge as it comes from the separators is washed with cold water, it becomes granular and coke-like in nature. Washing with cold water is effected by having the sludge propelled up an inclined trough by a spiral conveyor while water is sprayed over the length of the trough. This water then drains to sewers located at the lower end of the trough. The coked mass is delivered at the high end of the trough ready for disposal. If properly handled, the resulting coke may be mixed with coal and burned.

Naturaline Company Starts Activities

THROUGH the Naturaline Company of America, the Chestnut & Smith Corporation has concentrated its experimental work on sales and performance of finished motor fuels in one concern. The Naturaline Company of America has been formed by the Chestnut & Smith Corporation and its affiliated companies, all of which are manufacturers of natural gasoline. The company took its name from the name previously adopted for the finished aviation motor fuel offered by the concern.

The Naturaline Company of America is a closed corporation. Its officials are T. K. Smith, chairman of the board; E. I. Hanlon, president; D. E. Buchanan, vice president; J. H. Boyle, secretary-treasurer, and C. W. Anderson, sales manager. Headquarters are in the Chestnut & Smith building, Seventh and Cheyenne Streets, Tulsa.

Much of the sales promotional work is being conducted with the assistance of the five-passenger Buhl Airsedan, "Naturaline." This airplane is both an air office and an experimental airship on motor fuel performance. It is being used in connection with what the company is doing in co-operation with the Bureau of Standards. This activity is shown in the following announcement from the Bureau of Standards:

"The Naturaline Company of America is co-operating with the Bureau of Standards in its present study of the vapor pressure of aviation gasolines and has contributed a fund, which is being administered by the Society of Automotive Engineers, to pay the salaries of research associates engaged in this work."

Equipment to aid in this experimental work consists of a barometer, wet and dry bulb thermometers, flow meter and five thermo couples on the feed line between the gasoline tank and carburetor. These couples will record temperature changes in gasoline in relation to altitude. Such temperature changes in gasoline are part of the experimental work in connection with the study of the vapor lock, now under investigation by the Bureau of Standards.

Daily reports on this phase of the operation of the airplane will be sent to the Bureau of Standards to be checked with the laboratory findings on the subject.

The airplane will also be devoted to any other scientific study that is demanded.

The Naturaline Company of America will have foreign sales of the output of the several Chestnut & Smith manufacturing companies. A terminal has been built at Westwego, Louisiana, for loading tankers.

Tulsa.—C. W. Anderson is now sales manager of the Naturaline Company of America, having resigned from the sales department of the Marland Refining Company to take the new position. He had been with the Marland Company for five years, in the advertising department, later as assistant sales manager, and recently as manager of the company's Tulsa offices. Before joining the Marland organization, Mr. Anderson had been with the Indian Territory Illuminating Oil Company at Bartlesville.

The Naturaline Company specializes in production and sale of natural gasoline by-products and Naturaline for export and aviation purposes. The company was recently organized by the Chestnut & Smith Corporation, Brooks & Hanlon, Inc., Smith-Hanlon, Inc., and the Hanlon Gasoline Company; and it has built a plant at Westwego to manufacture Naturaline to supplement the output of gasoline plants of the parent companies.

In addition to its sales work, the Naturaline Company will take over the research work of the parent companies, continuing the experimental work which during the past three years has resulted in natural gasoline being recognized as an excellent aviation motor fuel. Private research work has demonstrated that the new fuels give satisfactory service under extreme conditions of high altitude and high as well as low atmospheric temperatures.

In line with this research work, the United States Bureau of Standards has begun an investigation of vapor pressures allowable for aviation gasolines, and natural gasoline companies expect a revision of present government standards.

Valves of Lasting Service

for all refinery jobs

FIGURE 23

An all semi-steel quarter-turn valve for use on hot tar and flow lines at temperatures less than 500° and pressures less than 250 pounds.

FIGURE 62

This Cast Steel Valve will handle safely and without giving any operating trouble, all refining jobs requiring a straight way valve, up to the ordinary temperature limits of refining operations. Especially recommended for vacuum work.

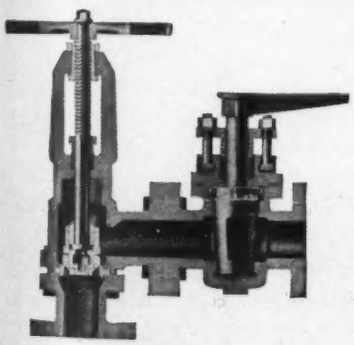


FIGURE 51

Simple, safe, convenient, this Hovalco-Homestead Blow-off Valve is helping many refineries secure better service from their boilers. Meets A. S. M. E. Code and all requirements of State laws. After using this valve four years, a refinery superintendent writes—"I don't think they are equalled by any valve made."

Homestead Valves, built on the famous "Quarter Turn" principle, are maintaining and widening their prestige among refiners. Under conditions that are fatal to valves of inferior quality or less adaptability, Homesteads are holding up for an average of not less than 10 years—usually without repair or regrinding the seats.

Homestead Valves have only one basic reason, other than their efficient design, for their marked success in refinery work. It is—*higher quality.*

We shall be glad to recommend the valve for any service where you have been having any valve trouble. The illustrations here cover only a few of the complete line which is described in a new catalogue. We would like to send you a copy.

FIGURE 2

A valve which may be cast in Acid Metal for use on agitators. The same pattern may be had in brass, suitable for any fluid that will not attack brass—and for temperatures not exceeding 400 degrees.

FIGURE 17

A Semi-Steel body Brass Plug Valve for which one company reports 12 years of satisfactory service from their initial installation in a Portland refinery. Suitable for 250 lbs. pressure in oil and gasoline lines and on storge tanks and gas lift installations.

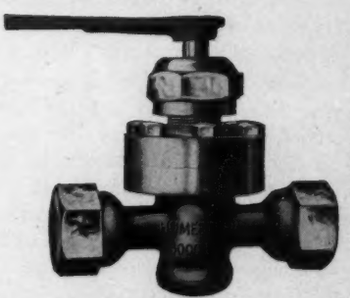


FIGURE 7-A

This high pressure Brass Homestead has been very successfully used by refiners on hydraulic lines and as blow-off valve on by-pass lines. It is quickly and easily operated and remains tight. Seats are unusually durable. Furnished for any pressures up to 3000 lbs.

Homestead Valves Sell on the Basis of Quality Service — Not on Price.

Homestead Valve Manufacturing Company, Homestead, Pa

HOMESTEAD

PRODUCTS: Quarter Turn Valves — Straight-Way Three-Way Four-Way Types — Full Round-Way Valves — Cast Steel Refinery Valves — Lubricated Valves — Hovalco Blow-Off Valves — Protected Seat Globe Valves B & O Pat. — Protected-Seat Hydraulic Operating Valves

Sulphur Compounds and Natural Gasoline

By HENRY COONEY

In the body of this manuscript Henry Cooney inserted this sentence:

"From a perusal of what follows it will be obvious that it was written by an operator and not a chemist."

It is pertinent to add that a gasoline plant operator does not need a diploma from a school of chemistry in order to apply the laws of chemistry to his work.

Mr. Cooney is with the Preston Oil Company at Sugar Grove, Ohio. What he has written here shows that he takes the findings of chemists and applies them to his work.

CONSIDERABLE interest is now being manifested in the question of sulphur compounds in natural gasoline. We are no longer certain "that all gasoline, except California, is non-corrosive," nor that all gasoline distilled from natural gas is free from sulphur or its compound. So the testing of natural gasoline for acidity, sourness and corrosiveness is becoming a part of the regular routine of tests being made at natural gasoline plants.

Serial No. 2843 of the Bureau of Mines gives the results of the doctor, corrosion and lamp tests on 130 samples of motor fuel collected during July, 1927. Each sample tested showed more or less sulphur but only seven contained more than the 0.10 per cent permitted by the U. S. government specifications for automobile fuel. Twenty-seven samples gave a positive doctor test (were sour), two samples were corrosive, two of the seven high sulphur samples were neither sour nor corrosive, yet they contained more than the permissible percentage of sulphur.

With the Federal Specification Board unwilling to permit more than 0.10 per cent of sulphur in the finished gasoline, the natural gasoline manufacturers will be expected to keep the sulphur content of their product well within this limit.

The following items on sulphur compounds and some methods of detecting them have been gathered from various sources, and are offered with the hope that they may be of interest to beginners in testing gasoline for certain classes of sulphur compounds, as sulphur dioxide, hydrogen sulphide, the mercaptans and some derivatives of the mercaptans.

Elementary sulphur is rarely found in natural gasoline, but if present it may produce corrosion, as sulphur combines with metals to form sulphides. These sulphides may change to oxides with the secondary result that sulphur is set free again. Another particle of metal is changed into a sulphide and the process starts over again.

Sulphur may be removed from a sample by washing with concentrated aqueous sodium sulphide, or it may be converted into a sulphide by the doctor solution.

Sulphur dioxide, SO_2, and its oxidation to sulphuric acid is a common cause of acidity, discoloration and corrosion in gasoline. The function of steam in distilling is to take up the SO_2. To accomplish this purpose, however, the steam should be air free.

We now come to a very troublesome sulphur compound, hydrogen sulphide, H_2S. Hydrogen sulphide is found in small quantities in many fields. H_2S is a colorless gas with a disgusting odor, and usually occurs with methane, ethane and other hydro-carbon gases. The specific gravity of H_2S is 1.1895 (air 1)—boiling point—79° F., hence it may be removed by weathering. It would follow then that a well stabilized gasoline will be free of this gas.

Most organic sulphur compounds will give off H_2S when heated and distillation shows that much of the sulphur in the oil will be obtained as H_2S in the gas.

Water at 62° F. absorbs 3.2 volumes of H_2S, so if the concentration is not too high this gas may be removed by washing in water. Where chemicals are used to remove H_2S, hydrated lime, soda ash, sodium hydroxide or the doctor solution as generally employed.

The test for H_2S is lead acetate, (sugar of lead). This lead acetate solution is made up by adding one teaspoonful of lead acetate to a quart of water, then enough acetic acid to make the solution clear. If H_2S is present a black precipitation of lead sulphide PbS, will be formed immediately on the addition of the lead acetate solution to the sample.

H_2S is a highly corrosive agent and leaks due to pitted fitting are often traceable to H_2S and its corrosive action.

There are two classes of compounds derived from H_2S—the hydro-sulphides, as ethyl hydro-sulphide, C_2H_5 SH; and the sulphides, as ethyl sulphide $(C_2H_5)_2S$.

Ethyl hydrosulphide, or ethyl mercaptan as it is usually called, is a colorless liquid with an offensive garlic like odor, boiling point 96.8° F. It has an hydrogen atom displaceable by a metal, hence it is corrosive. Other mercaptans closely resemble ethyl mercaptan in chemical properties. The mercaptans may be oxidized into sulphonic acids. These sulphonic acids contain the group SO_2OH, and are very corrosive acids.

Ethyl sulphide is a colorless, unpleasant smelling liquid boiling at 195.8° F. It is comparatively inert, giving a negative doctor test, when present in the gasoline, but gives off H_2S when heated and tends to be a corrosive factor.

Two salts of sulphuric acid may be mentioned, ethyl hydrogen sulphate $C_2H_5HSO_4$ and ethyl sulphate $(C_2H_5)_2SO_4$. Ethyl hydrogen sulphate is formed when ethylene is heated with sulphuric acid. It is corrosive. Ethyl sulphate is a neutral salt boiling at 406.4° F. decomposing slightly.

To summarize, we have seen that hydrogen sulphide may form mercaptans as C_2H_5SH or sulphides as ethyl sulphide, $(C_2H_5)_2S$. Then the mercaptans may be oxidized into the still more corrosive sulphonic acids. Further, these sulphur compounds may give off dydrogen sulphide when heated and start the cycle again. In addition the sulphur dioxide may be oxidized into sulphuric acid and that this sulphuric acid will attack many unsaturated hydro-carbons as ethylene starting another series of sulphur compounds.

Now, to make a few tests for hydrogen sulphide and other sulprur compounds we should obtain the following: lead acetate and acetic acid for the lead acetate solution, litharge and sodium hydroxide (lye) for the doctor solution, flower of sul-

A NEW ERA
IN NATURAL GASOLINE
is HERE
The highly competitive gasoline market has brought about many changes in the natural gasoline industry. Equipment and methods of only a few years ago are already obsolete. To obtain maximum recovery at a minimum of operating cost and a minimum of investment, progressive manufacturers are turning more and more to TULSA TYPE equipment.
TULSA TYPE

TULSA
Boiler & Machinery Co.
TULSA OKLAHOMA

Mention Where You Saw the Advertisement

The battle for business in the sales end of the oil industry demands every bit of thought and energy, every available dollar

How can the refiner succeed whose dollars are tied up in storing excessive fuel oil and who is burdened with the problem of somehow getting rid of this unwanted surplus?

But the refiner whose plant is equipped with Dubbs Cracking Process with Flashing

System, is "stripped for action" because he is able, in one operation, to convert *all* of his cheap fuel oil, as rapidly as he produces it, into super-quality premium gasoline and premium fuel oil or (with the non-residuum type of operation) all into super-quality gasoline and coke

Universal Oil Products Co
Chicago, Illinois

Dubbs Cracking Process
Owner and Licensor

phur, copper strips or better, a copper dish, and a copy of Technical Paper 323B of the Bureau of Mines.*

First test for H_2S by adding lead acetate solution to a sample of gasoline. A black precipitate of lead sulphide Pb.S is formed. This gasoline needs further weathering, washing with water, or a light chemical wash. The doctor solution gives the same black precipitate as the lead acetate solution, but the doctor solution will also react with other sulphur compounds, if present; hence the results would not be the same.

The sample may not contain H_2S or we may filter out the lead sulphide if it should, and have a sample free for H_2S to test for mecaptans.

The doctor or sodium plumbite solution is generally used for neutralizing the mercaptans. This doctor solution is prepared from litharge, PbO, and sodium hydroxide, Na_6H, as above stated, see Technical Paper 323B for the directions for preparing this solution, and interpreting the tests made with it.

Add the doctor solution and sulphur as directed by this Technical Paper, "A yellow color, turning to orange or brown indicates mercaptans," also a yellow precipitate only "indicates mercaptans—no hydrogen sulphide present to produce grey or black discoloration of the sulphuric film.

The chemical change that has taken place has been outlined as follows: ethyl mercaptan being present.

*Technical Paper 323B is obtainable from the Superintendent of Documents, Washington, D. C. Price 15 cents.

$2C_2H_6$; $SH + Na_2\ PbO_2 = (C_2H_5S)^2 + 2\ Na\ OH$

Ethyl mercaptan plus solution equals lead mercaptan plus sodium hydroxide.

The sulphur is now in the lead mercaptan.

Sulphur is now added.

$(C_2H_5S)^2\ Pb) + S = C_2H_6 + Pb_5$

Lead mercaptan plus sulphur equals ethyl disulphide plus lead sulphide.

The ethyl disulphide remains soluable in the gasoline but the lead sulphide settles out. No sulphur has been removed but the gasoline is sweet to the doctor test, and should settle clear. "When a sample settles rather slowly, but definitely clear... it is not likely to show excess sulphur, and on the other hand most surely has enough."

Make the copper strip test. If the strip is not discolored our gasoline is non-corrosive at that temperature and approaches 100 per cent. There is a possibility, however, of a gasoline containing too much sulphur by the lamp or similar test and yet be "sweet," and non-corrosive as before stated. This is due to comparative inert sulphur compound that are not acted upon by the doctor solution.

To actually reduce the sulphur content may require treatment with sodium sulphide, sulphuric acid, blue vitriol, etc.

The tests for acidity and gum in gasoline are given in Paper 323B and should be conducted along with the other tests mentioned for an acid or a gum ladened gasoline may give a positive doctor test.

Methods Applicable to Determination of Sulphur

By RALPH E. GOODE

SULPHUR and its compounds which are present in most pertoleum oils in varying quantities are generally precipitated as barium sulphate by most methods used in the laboratory. The presence of sulphur and its compounds may be detected qualitatively in several different ways. In the case of heavy oils the Emerson and Parr Calorimeter Bombs are used most frequently for determinating the percentage of sulphur. The A. S. T. M. Sulphur Lamp is used for the determination of the percentage of sulphur in light oils.

The procedure followed with both types of Calorimeter Bomb is practically the same, the Emerson apparatus perhaps being more frequently used. In testing for the content of sulphur with the Emerson apparatus the method used is as follows: One gram of the sample of oil, coke or coal is weighed out on the balance with an accuracy of one-tenth of a milligram. If a light oil is to be tested it is placed in a sulphur free gelatin capsule at the beginning of the test to prevent loss by evaporation and when placed in the pan of the bomb chemically pure benzoic acid is sprinkled upon it to start combustion at the time of firing. The iron wire used for firing the material must be of approximately 0.32 gage and chemically pure. The bomb is closed after the adjustment of the terminals and after 10 c.c. of distilled water is placed in the lower half of the bomb, oxygen is introduced slowly into the bomb until the pressure reaches 300 pounds. The valve is then closed and the bomb placed in a bath of water and ignited.

Upon opening the bomb after the pressure is released through the valve and the bomb is sufficiently cool the sulphuric or sulphurous acids which formed during combustion are washed out into a beaker by means of a wash bottle. The washings are then made alkaline with strong ammonia and then boiled to precipitate any metals. The precipitate, together with any ash which may have resulted during combustion, is then filtered off and washed with hot distilled water. The filtrate is then acidified with hydrochloric acid, a few drops of bromine water (5 drops) added and boiled. Hot barium chloride is then added and the precipitate is allowed to settle. The precipitate is filtered off and washed with hot distilled water until free from chlorides. It is then placed in a platinum crucible, dried slowly, ignited and weighed. The percentage of sulphur is obtained by multiplying the weight of barium sulphate by .137 and dividing by the weight of the original sample taken.

In testing for the percentage of sulphur in light oils such as pressure distillate, naphtha and illuminating oils the A. S. T. M. method as outlined in Bulletin 323-B, Bureau of Mines is followed. This consists essentially in absorbing the sulphur compounds in pure sodium carbonate by means of suction through the apparatus and titrating the solute with a standard solution of hydrochloric acid. The main difficulty encountered in this method is in getting the proper flame from the oil, free from carbon and in good burning qualities. Great care must be exercised in the selection of the cotton wicking as the ordinary kind will not give a proper flame and one that will continue to burn. Good results may be obtained by using a wicking made of two strands of absorbent cotton passed parallel through the wick tube. Sulphur determinations with this apparatus check with those made with the Emerson within a range of .03%.

The rapid determination of sulphur in coke from the cracking plant may be made as follows: The sample is dried, pulverized and screened through a 150 mesh sieve. One half a gram of the coke is carefully mixed with 12 grams of sodium peroxide, three-tenths of a gram of benzoic acid, and three-

tenths of a gram of powdered potassium chlorate. The mixture is then placed in a suitable nickel crucible (Pennock and Martin) which is covered tightly and placed halfway in a bath of cold water. The contents are then ignited with an incandescent wire. Care being taken that fusion results without violence and the melt should be free from carbon or unburned coke.

Allows the crucible to cool and then place it in a 500 c.c. breaker and dissolve the fusion with a small amount of distilled water. Make the solution just acid with hydrochloric acid and filter the insoluble. Wash the paper once and add 15 c.c. of saturated bromine water and boil until the excess bromine is expelled. Then add 10 c.c. of a 10% solution of barium chloride and without stirring boil for five minutes. Allow the barium sulphate to settle out and filter through a fine filter paper and wash with hot distilled water until entirely free from chlorides which may be detected with silver nitrate. Place the filter paper and contents in a vitreosil or platinum crucible and burn very slowly for a time. When the paper is gone to an ash, ignite strongly, cool and weigh as barium sulphate. The weight of barium sulphate multiplied by .137 and 200 gives the percentage of sulphur in the coke.

For ordinary purposes a determination for sulphur may be made as follows: Grind the sample and weigh out approximately 2.5 grams in a flat dish, dry to a constant weight at a temperature no higher than 221° F. The loss may be reported as moisture. Then dissolve the residue from the moisture determination in carbon bi-sulphide. Filter through a weighed Gooch crucible which has been previously treated with a small amount of carbon bi-sulphide. Then wash the residue with a small amount of carbon bi-sulphide and again with a small amount of ether, then dry and weigh. Subtract from the total amount the percentage insoluble in carbon bi-sulphide and the percent of moisture and the result will be the percentage of sulphur which is insoluble in carbon bi-sulphide.

Sulphur as indicated qualitatively in painter's naphthas and other paint thinners is detected by a thorough mixing of a sample of the naphtha with enough pure white lead to form a creamy paste. Spread this upon a clean, dry watch glass and heat in the electric oven to a temperature of 250° F. for approximately two hours. Sulphur when present will darken the color of the white lead paste to a black or brown.

A reliable and standard test for hydrogen sulphide where present in large or small amounts is made as follows: Take a crystal of lead acetate and dissolve it in a test tube containing a small amount of water. Saturate a piece of filter paper with the solution. Thereafter pouring off as much as possible of the liquid that covers the sulphide solution, add a little concentrated hydrochloric acid to the test tubes containing them. Then place the wet acetate paper over the mouths of the tubes. Expose the paper to the vapors arising from liquid under test. The glistening, black film that forms is lead sulphide.

Exposition Will Have Latest Plant Equipment

APPARENTLY chief interest of the International Petroleum Exposition and Congress, from the viewpoint of the refining and natural gasoline divisions of the petroleum industry, will be in the numerous exhibits of improved plant equipment.

The exposition will be held this year, October 20 to 29, in Tulsa. The site is the same as that of the 1927 exposition. The date has been set in October in order to escape the usual weather change of September.

More space has been sold for the 1928 exposition than for any previous affair. Reservations passed 200 prior to September 1, and indications are that 250 concerns will have equipment on display. Considerable of this will be equipment for refineries and natural gasoline plants. The manufacturing division of the industry witnesses thousands of instances of new and improved equipment each year. This fact alone assures the opportunity of inspecting something that is modern in actual plant operation.

As in past years the exposition will devote a share of its program to safety work. The foremost part of this will be the annual first aid meet of the Safety Section of the Mid-Continent Oil & Gas Association. The United States Bureau

(*Continued on page* 100)

EXPOSITION OFFICIALS INSPECT GROUNDS

Left to right—B. E. Horrigan, A. F. Bourne, William Holden, Alf G. Heggem, W. G. Skelly, president; D. D. Wertzberger, Frank Hinderliter, J. M. Post, W. M. Bovaird, J. Burr Gibbons, general manager; Brooks Gatelius, William Schlueter, Charles P. Dimmit, Clyde Alexander, A. W. Leonard and P. D. Hayes.

NEW EQUIPMENT, CATALOGS, BULLETINS, BOOKS

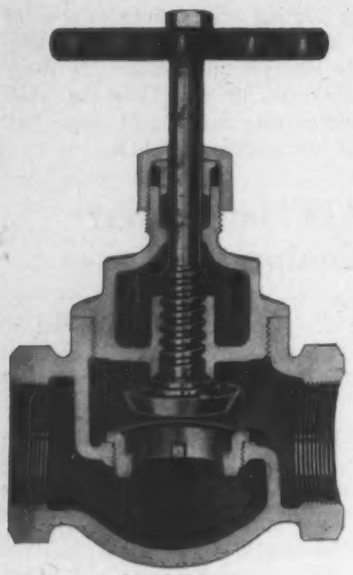


FIGURE 1

New Replate Valve

CENTRAL VALVE MFG. CO.

Central Valve Manufacturing Company, 435 N. Michigan Avenue, Chicago, announces a new valve, the Replate, made in globe, angle and Y bodies. The valve is of rugged construction and is manufactured for both high and low pressure service.

The Replate valve in section is shown in Figure 1. In this design an outstanding feature is that the head of the stem is capped with a removable cold rolled nickel plate, formed to give a ball joint effect, and fully supported by the end of the stem. This head plate, working against a Monel metal removable seat, offers sufficient variation in the hardness of the two metals to make a complete bearing and a tight joint. Being somewhat softer, the nickel head plate takes most of the wear and has been designed so that it can be replaced readily without removing the valve from the line.

In an emergency such as would exist where an extra plate were not available, the end of the stem has been designed to function as a head plate. Repacking may be done under pressure in the wide open as well as the closed position, as a complete joint is formed between the bonnet and the head of the stem. A dead air space in the bonnet prevents rapid transmission of heat from the body to the handle. The short stem and bonnet reduces to a minimum the space required for installation.

As shown in Figure 2, the feature of the Perfection self-grinding valve is the use of a short spiral spring and a spring block threaded to the valve stem, which forces the valve to its seat, so that when the valve is being closed, it revolves and pulverizes and removes any dirt or scale on the seat. This action tends to insure a constantly clean valve seat in spite of severe working conditions. As the stem comes to a seat in the body, the threaded section of the stem being engaged with the threads in the spring block, the latter moves upward on the stem against the spiral spring. The pressure of this spring against the block holds the valve to its seat. Further rotation of the stem brings the spring block solidly against its upper stop in the bonnet and the valve to a firm seat. Meanwhile the stem has been revolving on its seat and has ground itself to a tight joint. Where additional grinding is necessary, a short movement of the handwheel back and forth in the closed position is sufficient.

A shoulder on the head of the stem makes a tight joint on the inside face of the bonnet when the valve is in a wide

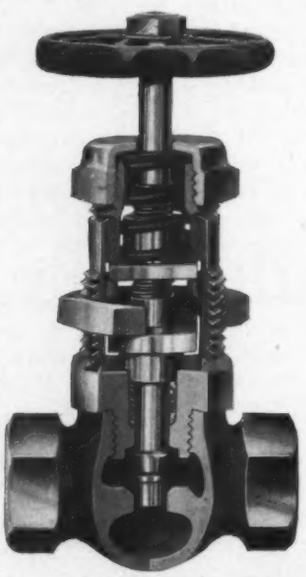


FIGURE 2

open position, allowing for repacking under pressure in both closed and wide open positions. As the stem threads, spring and spring block are located outside the bonnet above the packing, they can be lubricated easily. To control the grinding pressure on the seat according to the varying operating pressures and hardness of the impurities encountered, the spring retaining cap at the top of the bonnet may be adjusted up or down within a definite range. These valves are intended for working pressures to 300 pounds gauge and temperatures to 550 degrees F.

More information may be obtained by writing the company.

The Power Plant Equipment Company, 1737 Walnut Street, Kansas City, Missouri, announces a catalog on Marley Spray Cooling Equipment. In addition the publication contains much cooling data along with other valuable information to engineers dealing with cooling problems. The catalog is described as a handbook on spray cooling. Copies may be obtained by writing the company.

Sarco Company, 183 Madison Avenue, New York City, has published bulletins on Sarco Traps for Process Steam and Sarco Combination Float and Thermostatic Steam Traps. Copies of both may be obtained by writing the company.

R. E. Mossman, former assistant superintendent of The Texas Company refinery at Casper, Wyoming, has been promoted to the position of superintendent. Pendelton Beall, former engineer at the Casper plant, will be superintendent of the company's new refinery to be constructed at Cody.

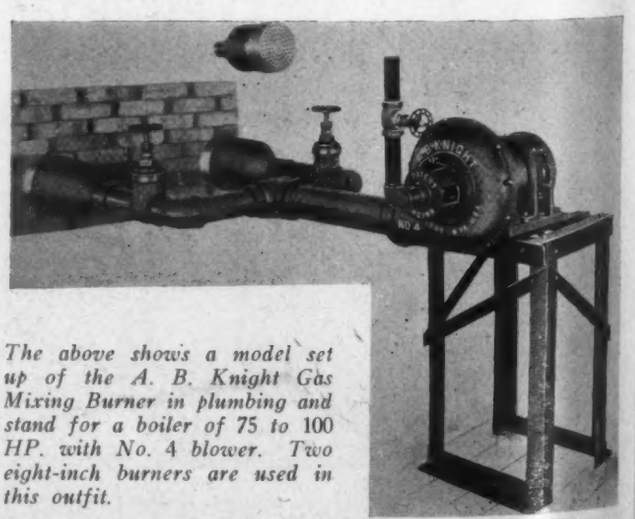
Gas Mixing Burner

A. B. KNIGHT

A. B. Knight, 117 East Park Avenue, Fairmont, West Virginia, has on the market a new air and gas mixing burner and blower.

The blower part of the outfit is operated by a small motor. The principal feature of the blower is the one-piece aluminum fan with a large number of small holes in its center through which the gas comes. The intake fuel pipe is connected to the hub of this fan. It is claimed that as the gas leaves the fan through the many holes, a perfect mixture is obtained. An adjustable air shutter controls the air supply.

Further information may be had from the company by request.



The above shows a model set up of the A. B. Knight Gas Mixing Burner in plumbing and stand for a boiler of 75 to 100 HP. with No. 4 blower. Two eight-inch burners are used in this outfit.

Let

Solve Your Packing Problems

Pride in achievement means more to many men than accumulation of large fortunes.

As the inventor of modern packing, I bought the controlling interest in the Crandall Packing Company so that I could establish a standard of quality and service unequalled.

I invite correspondence from manufacturers, large and small, who may have packing problems. In those matters that can be handled by correspondence, I will gladly give my personal attention.

Crandall Service-Salesmen are always at your service. You place yourself under no obligation when you ask the help of any member of my organization.

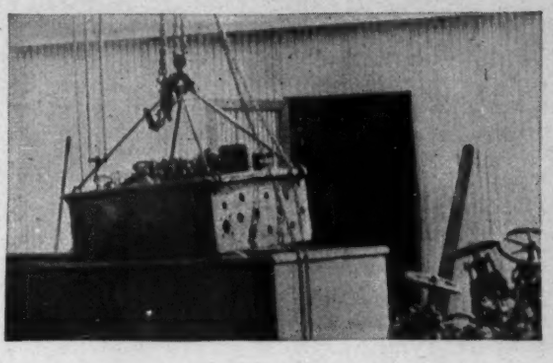
At this time, I would deeply appreciate your request for a sample of Luball, the new and better lubricant.

O. J. Garlock

Crandall Packing Co.

Perfected Packings for Every Purpose

Palmyra, N. Y., Boston, Chicago, Cleveland, Houston, New York, Pittsburgh, Philadelphia, Kansas City, Mo., St. Louis



Ready for a quick clean-up

THIS basket of dirty parts—valves, fittings and tools—is all set for a quick, thorough cleaning. Oil, grease and muck accumulations begin to move the instant it is lowered into the Oakite cleaning solution. A short soak and the job is done—automatically—with no more effort than lowering and raising the parts.

There is a similar easy Oakite method for doing every cleaning operation in the oil industry. Storage tanks, tank cars, drums, trucks and dirty refinery equipment are cleaned with corresponding time and labor savings. *Safely,* too, because there is no fire hazard with Oakite materials. They are non-inflammable and non-explosive, being used in water solutions.

Get in touch with our nearby Service Man for further details. Drop us a line and he will call with all the facts about this better way to clean.

Manufactured only by

OAKITE PRODUCTS, INC., 50B Thames St., NEW YORK, N. Y.

Oakite Service Men, cleaning specialists, are located at

Albany, N. Y., Allentown, Pa., *Atlanta, Altoona, Pa., Baltimore, Birmingham, Ala., *Boston, Bridgeport, *Brooklyn, N. Y., Buffalo, *Camden, N. J., Canton, O., Charlotte, N. C., Chattanooga, Tenn., *Chicago, *Cincinnati, *Cleveland, *Columbus, O., *Dallas, *Davenport, *Dayton, O., *Denver, *Des Moines, *Detroit, Erie, Pa., Fall River, Mass., Flint, Mich., Fresno, Cal., *Grand Rapids, Mich., Greenville, S. C., Harrisburg, Pa., Hartford, *Houston, Texas, *Indianapolis, *Jacksonville, Fla., Joplin, Mo., *Kansas City, Mo., *Los Angeles, Louisville, Ky., Madison, Wis., *Memphis, Tenn., *Milwaukee, *Minneapolis, *Moline, Ill., *Montreal, Newark, N. J., Newburgh, N. Y., New Haven, *New York, *Omaha, Neb., Oshkosh, Wis., *Oakland, Cal., *Philadelphia, Phoenix, Ariz., *Pittsburgh, Pleasantville, N. Y., Portland, Me., *Portland, Ore., Poughkeepsie, N. Y., Providence, Reading, Pa., Richmond, Va., *Rochester, N. Y., Rockford, Ill., *Rock Island, *San Francisco, *Seattle, Springfield, Mass., South Bend, Ind., *St. Louis, *St. Paul, Syracuse, N. Y., *Toledo, *Toronto, Trenton, *Tulsa, Okla., Utica, N. Y., *Vancouver, B. C., Williamsport, Pa., Worcester, Mass.

*Stocks of Oakite materials are carried in these cities.

OAKITE

TRADE MARK REG. U.S. PAT. OFF.

Industrial Cleaning Materials and Methods

(*Continued from page 97*)

of Mines is again co-operating in this event. This year the first aid meet will have the backing of the Petroleum Division of the National Safety Council.

The first aid meet will be held the afternoon of October 22 at the Exposition grounds. There will be prizes for the members of winning teams. It has been decided to present seven medals for each team, instead of six, as in the past. This will allow the substitute of each team to have a medal, identical with those given team members.

The majority of competing teams will come from refining plants and from natural gasoline plants, although the meet will be open to the whole petroleum industry.

A departure this year will be the safety banquet, which will be held the evening following the first aid meet. All prizes are to be presented then. An effort will be made to have the members of all competing teams attend this banquet.

The 12 standing committee of the Natural Gasoline Association of America will meet during the exposition.

Other organizations that will take advantage of the exposition to have meetings are the Mid-Continent Oil & Gas Association, the American Institute of Mining and Metallurgical Engineers and the American Association of State Petroleum Geologists.

The exhibits of the technical building will bear on refining in many instances. These exhibits, many of which will be fostered by organizations, are being placed under the supervision of a technical committee.

The exposition of 1928 will be the largest ever attempted. Grounds and buildings have been enlarged by the expenditure of $45,000. Much of this improvement is in concrete walks, improved roads and parking places. Other comfort and convenience assets are being added to the exposition plant.

Many of the refining concerns having headquarters in Tulsa are taking advantage of the exposition in order to have plant employes at headquarters for meetings and conferences. While these are of company nature, they will assure enlarged attendance.

Cody, Wyoming.—The work of grading for the tanks of the Texas Corporation, and the laying of tracks from the Burlington railroad for the new refinery has already been started. There will be two tracks, one into the refinery buildings for handling equipment and material and the other for loading racks. The site is on a 260-acre tract acquired recently from the government and east of the Burlington station. It lies between the railroad tracks and the Shoshone river and north of the corporate limits of the town. Orders have been placed for material and equipment and as soon as the railway tracks have been completed, supplies will start moving in.

A new $250,000 hotel is to be constructed in Cody, the first two floors being reserved for stores and offices, and the upper three for 95 modern hotel rooms. The hostlery is to be completed by spring of next year.

The Cody Club and the state highway department are building a better and shorter road to the Oregon Basin field which will greatly facilitate traffic, particularly during the bad winter weather.

Riverton, Wyoming.—The New York Oil Company is tearing down its absorption plant here and moving it into the Sand Draw field, southeast of here. The New York recently cancelled its contract with Teton Gas Product Company, carbon black manufacturers, and for this reason the Teton company will also have to dismantle its plant and move it elsewhere.

New York.—The Standard Oil Company of New Jersey's program of improvement at its Bayway refinery at Elizabeth, New Jersey, will include two double unit cracking installations of the tube and tank type. The new units will have a capacity of 14,000 barrels of gas oil daily. They are expected to be completed early in 1929.

Four-Unit Heat Exchangers

HEAT EXCHANGERS

produce remarkable savings in fuel and cooling water

Single and multiple-unit types. Designed in every case to meet specified operating conditions.

We are equipped to build heat exchangers of large capacities. Send for Bulletin 12-H

Also jets, valves, condensers, spray nozzles, vacuum pumps, gear pumps, and oil burners for the oil industry. Send for illustrated booklet showing entire line of refinery equipment.

SCHUTTE & KŒRTING Co.

1253 N. 12th St., PHILADELPHIA, PA.

DEPENDABLE REFINERY PUMPS

Because of their recognized dependability, ruggedness of construction and efficiency, Byron Jackson pumps have been adopted by many refineries.

Throughout the oil industry, the use of Byron Jackson equipment is regarded as insurance against interrupted operation.

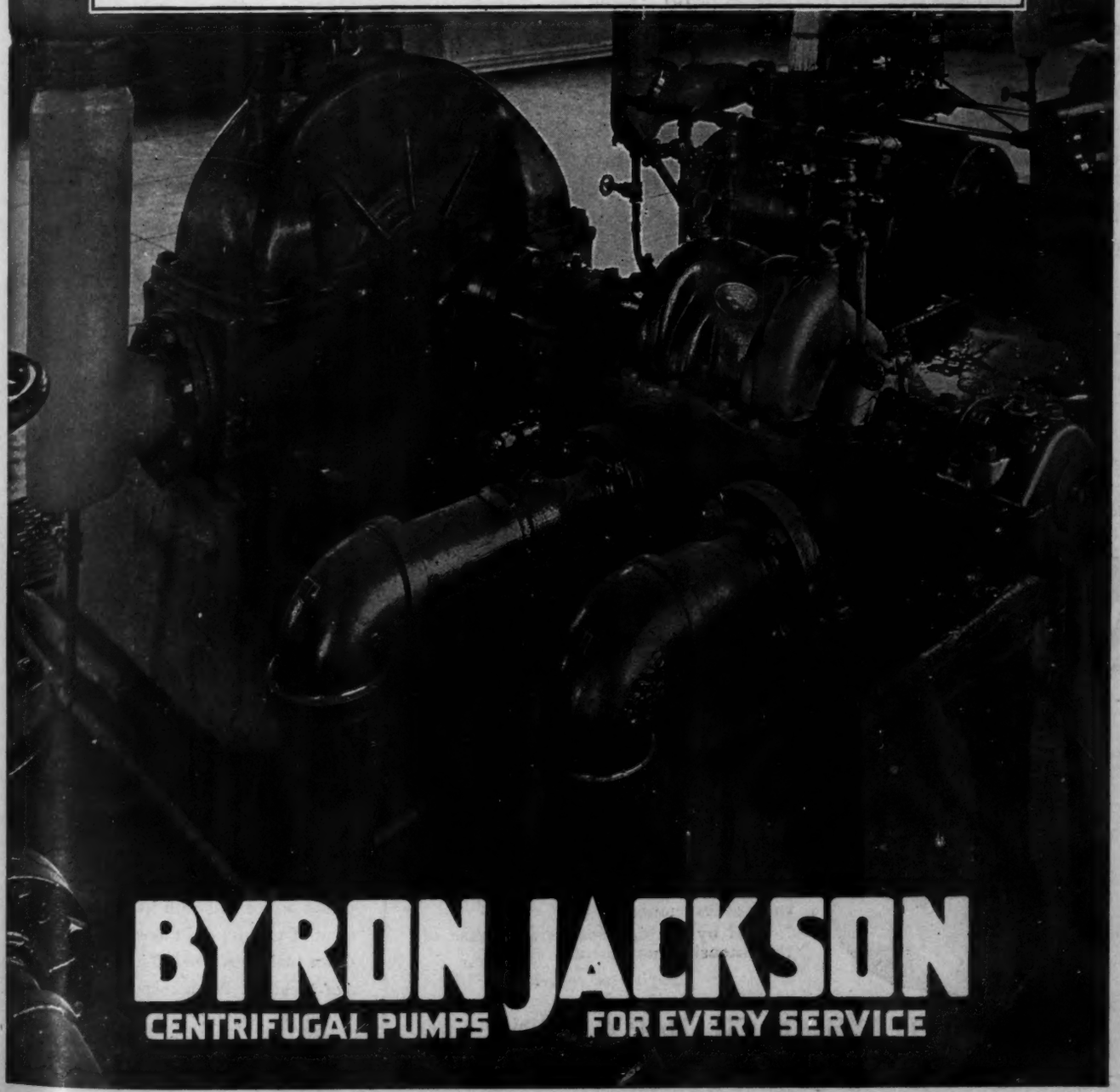
Back of every Byron Jackson pump is more than fifty years of experience in building special equipment for special service.

SINCE 1872

BYRON JACKSON PUMP MANUFACTURING CO.

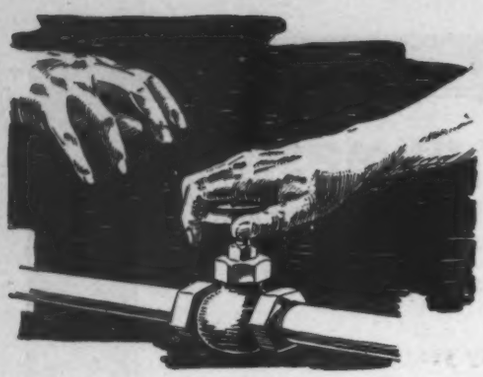
FACTORIES: BERKELEY, LOS ANGELES, VISALIA

Branches: San Francisco, Portland, Salt Lake, Dallas, Phoenix



BYRON JACKSON

CENTRIFUGAL PUMPS FOR EVERY SERVICE



Who Turns the Handle in *Your* Plant?

You win—or you lose—depending upon how the hand valves are operated in processes requiring heat treatment. Products damaged or ruined by too much or too little heat costs industry a tremendous toll. And this loss is due entirely to expecting human beings to do the ***impossible***—to "guess" temperatures accurately.

Mechanical control, on the other hand, takes the guess out of temperature control. Once set for a given temperature, the Sarco Temperature Regulator maintains exactly that temperature without attention. It can't make a mistake.

The Sarco is inexpensive to buy and to install. No pipe lines needed. It is entirely self-contained. Requires no compressed air, water pressure or electricity to operate. It is also inexpensive to maintain.

30-DAY FREE TEST

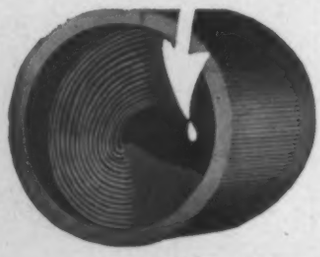
Send for a Sarco and try it for 30 days. If it doesn't do your work as you expect, send it back and you won't owe us a cent.

Write for free Booklet S-96

SARCO COMPANY, INC.
183 MADISON AVE., NEW YORK CITY
Boston, Buffalo, Chicago, Cleveland, Detroit, Philadelphia, Pittsburgh, St. Louis
Pencock Bros., Montreal

SARCO TEMPERATURE REGULATOR

The Spiral Motion Is the Trick



That's why Hood's Chemico Spiral Packing Rings have been selected by so many engineers. The center hole prevents choking and clogging.

Made of acid-resisting shale—practically indestructible.

Used by all big producers because of the intensive movements.

Samples on request.

B. MIFFLIN HOOD CO., DAISY, TENN.

Jackson Moves Main Office

The Jackson Engineering & Equipment Company of Los Angeles, representatives of the Fluor Construction Company and of Heat Transfer Products, Inc. (Petroleum Division) recently announced the removal of their office from 909 East 59th St., Los Angeles to 1022 Pacific National Bank Building in the same city. The new office will be under the management of Mr. Karl Finsterbusch who has been in charge of the company's Pacific Coast business for the past year. Inquiries regarding cooling towers, heat transfer equipment, absorption plants and construction service will be promptly and capably handled when addressed to the new office. Other offices maintained by this company are at 90 West Street, New York City, 2015 Magnolia Building, Dallas, Texas, and 502 National Bank of Commerce Building, Tulsa, Oklahoma.

Machinery Company Organized

The D. S. Mair Machinery Company, 505 Esperson Bldg., Houston, Texas, has been formed by D. S. (Stanley) Mair, to handle Machine Tools and accessories. Mr. Mair has been for 17 years with the Jos. T. Ryerson and Sons Company as branch manager. This organization is familiar with the requirements of the oil industry in the way of machine tools, etc.

Mr. Mair states that a complete line of Landis Chasers, made by the Landis Machine Company of Waynesboro, Pennsylvania, is carried in stock in Houston. The D. S. Mair Machinery Company are the representatives for Landis Machine Company in Texas.

Shreveport, Louisiana.—At a cost of about $250,000, the Shreveport Producing & Refining Company has made extensive improvements and enlargements at its Jewella plant, Shreveport; and as a result the capacity of the plant will be increased over 7000 barrels, raising it to around 12,000 barrels a day. New equipment includes a pipe still with its auxiliary equipment of pumps; eight new 1500-barrel rundown tanks; two 80,000-barrel crude storage tanks; a 244-horsepower boiler; addition to loading rack capacity; a new compressor unit; pumps, and heat exchangers.

EVERY ENGINEER KNOWS
WHAT A GREAT INDUSTRIAL LEADER DID NOT
"That Steam Should be Purified"

"What?" he said, "I thought all steam was pure of itself—I didn't know it had to be cleaned."

Every engineer admits the desirability of clean, dry steam and its beneficent and economical effect in use.

All are not agreed, however, on the methods to be employed to achieve this condition.

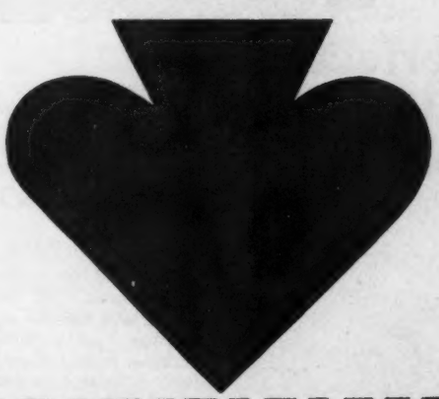
Complete purification is necessary—partial purification is useless.

These things are discussed in an interesting way in the bulletin entitled

"A CUBIC FOOT OF STEAM VISUALIZED"

It is free to engineers and others who deal with steam problems.

USE THE COUPON



ANDREWS-BRADSHAW COMPANY division of BLAW-KNOX COMPANY **605 Farmers Bank Bldg., Pittsburgh, Pa.**

Please send booklet entitled "A CUBIC FOOT OF STEAM VISUALIZED"

*Name*_____

*Street*_____

*City*_____

Mention Where You Saw the Advertisement

Patented
R. S.
Alligator
Oil Thief

New Improved

Alligator Oil Thief

3-inch Quick Opening Trap or
2-inch Quick Opening Trap
(No Tools Required)

Can't Stick
No Clogging
Easily Adjusted

SAMPLES accurately taken at any depth by use of GAGE POLE or CHAIN.

12-inch OIL THIEF adjustable for bottom settlings samples up to 21 inches. Rugged construction.

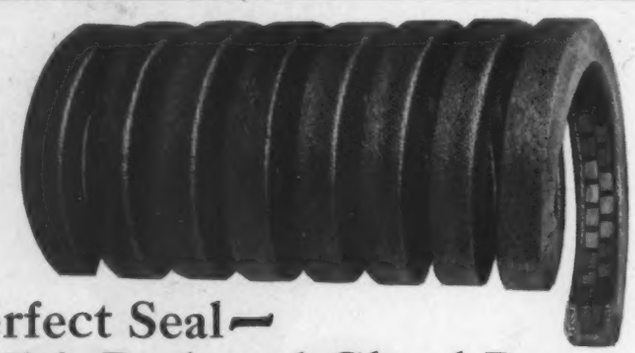
Manufactured by

The Refinery Supply Company

Largest Scientific Material House in the Southwest

TULSA, OKLA.
Fourth and Detroit Sts.

DALLAS, TEXAS
3404-06 Main Street



A Perfect Seal— With Reduced Gland Pressure

SOME packings are kept tight only by constantly increasing the gland pressure.

But not "V" Pilot!

Because of its resilient asbestos back, "V" Pilot expands and sets the metal to the rod, requiring reduction, rather than increase, in gland pressure until the perfect seal is obtained with the minimum of friction.

Not only does this "V" Pilot feature mean less wear on the rod or plunger—it is also a very definite assurance of longer packing service.

You can more generally use "V" Pilot throughout the refinery and gasoline plant than any other packing. Give "V" Pilot a trial. Get in touch with the nearest distributor—or if you wish, write us for prices and complete information.

Send for a copy of this new bulletin containing data on the applications of "V" Pilot in the oil industry.

THE NEW JERSEY ASBESTOS COMPANY
One Water Street, New York, N. Y.

Philadelphia, 216 Walnut St. — Baltimore, 17 So. Gay St. — Norfolk, 111 E. Main St. — San Francisco, 37 Spear St. — Wilmington, Cal., 236 Avalon Blvd. — Boston, 148 Pearl St.
Texas Rubber Specialty Co., Houston, Texas
Norvell-Wilder Hardware Company, Beaumont, Fort Worth, Shreveport

"V" PILOT PACKING

Phillips Plant to Handle Hydrogen Sulphide Gas

Bartlesville, Okla.—Construction work has started by the Phillips Petroleum Company on a new natural gasoline plant of special design, located in the Church and Fields oil pool, near McCamey, Upton County, Texas, which will be the largest plant of this kind ever built.

The plant is designed to handle approximately 50,000,000 cubic feet of gas per day and to produce 75,000 gallons of natural gasoline every 24 hours. The gas in this area, while rich in gasoline content, contains large quantities of hydrogen sulphide, a poisonous gas, and the plant has been designed especially to handle this difficult situation from a manufacturing standpoint and for the protection of employes.

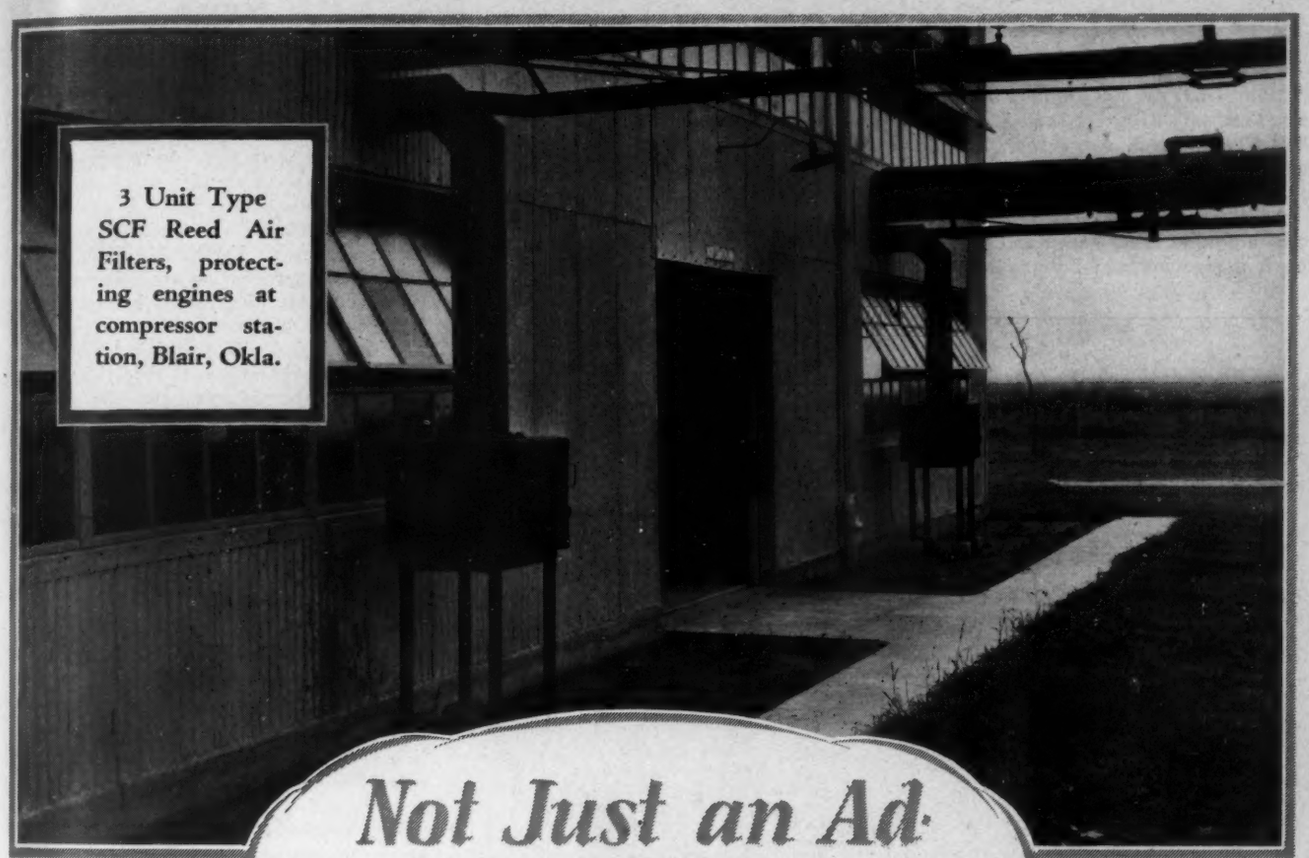
In addition the gas is corrosive and the parts of the plant where the gas comes into contact with metal will all be made from alloy metals other than steel. Extensive experiments by the company with this type of gas and in corrosion will cause the use of some entirely new materials, new design and construction methods.

The plant is located about halfway between the towns of McCamey and Odessa in Upton County, and will be called the McCamey plant. Shipments of the finished product will be made by a 25-mile pipe line to McCamey. The plant should be running some time in December, according to Phillips officials. Motive power for the manufacturing operations will be furnished by twelve 400 horsepower gas engines, while other parts of the plant will be commensurate with its size and the amount of daily production.

G. M. Jackson, president and general manager of the Jackson Engineering & Equipment Company of Los Angeles, recently returned to that city after a six months' absence in New York and the Mid-Continent, during which time additional arrangements were concluded for the distribution of Fluor Cooling Towers in all Eastern territory. A considerable portion of Mr. Jackson's time was spent with the engineers of Heat Transfer Products, Inc., of New York, in developing a standard line of heat exchange equipment for the petroleum industry, which will be distributed through the Jackson Engineering & Equipment Company. This company has recently opened a new office in Room 509, 90 West Street, New York City.

Tulsa.—Proposing to have each in operation about September 15, the Thermal Oil & Gas Company of Tulsa is building two 5000-gallon natural gasoline plants in Oklahoma. One is located between Bristow and Slick in Section 13-15-9, and the other is about four miles east of Wewoka in Section 1-8-8.

REED AIR IS CLEAN AIR



3 Unit Type SCF Reed Air Filters, protecting engines at compressor station, Blair, Okla.

Not Just an Advertising Slogan-but a Scientific Fact

The Reed principle of scientific air filtration insures:

1. *Highest Efficiency in Dust Removal:* because the crimped-wire filtering media used in Reed Filters exclusively, divides air into innumerable fine streams and brings it into positive contact with the adhesive coated filaments.

2. *Lowest Operating Cost:* because the graduated density of the media prevents restriction of air flow and increases resistance from excessive dust accumulation on the face of the filter.

3. *Largest Dust Capacity:* because Reed crimped-wire offers the maximum amount of adhesive coated surface for holding the dust—over 1500 square feet in each filter cell.

REED AIR FILTER CO., INC.
LOUISVILLE, KY.

Distributed by

SMITH SEPARATOR COMPANY
Tulsa Okla.

HOUSTON FORT WORTH SHREVEPORT LOS ANGELES

In view of the above advantages, Reed Air Filters have become the preferred means of keeping dust laden air out of compressors and engines in hundreds of installations throughout the Oil Industry?

A Smith Separator Company representative will gladly explain in detail how you may obtain clean air at minimum cost with Reed Air Filters.

Send to Smith Separator Company,
Box 2003, Tulsa, Oklahoma,
for Illustrated Catalogue

Reed Air

TRADE MARK

filters ALL METAL

Abstracts of Technical Literature
Of Particular Interest to the Plant Operator

Cracking

Cracking, Especially of Lignite Tars and Shale Oils, by the Blumner Process. Karsten. (*Oesterr. Chem.-Ztg.*, 39-9 (1928) *C. A., Vol. 22, No.* 11, *pp.* 2050-51.) A description is given of the Blumner process, German patent 338,846 (1922.) Crude oil, shale oil, low-quality lignite tars or tars of any kind are pumped and injected through a pipe lowered into molten lead contained in an autoclave, heated externally. The charged oil bubbles to the surface of the melt without touching the walls of the heated zone. The process is carried on under pressure of 35-40 atmospheres at about 450 degrees C., the cracking temperature of the oil. Automatic circulation of the melt causes constant heat transfer from the source of the oil, thus preventing overheating and coking. Advantages of the process are: larger yield of benzine of better quality, uninterrupted operation, greater safety and ability to handle crude and distillates simultaneously. The process is operated commercially in a plant near Berlin.

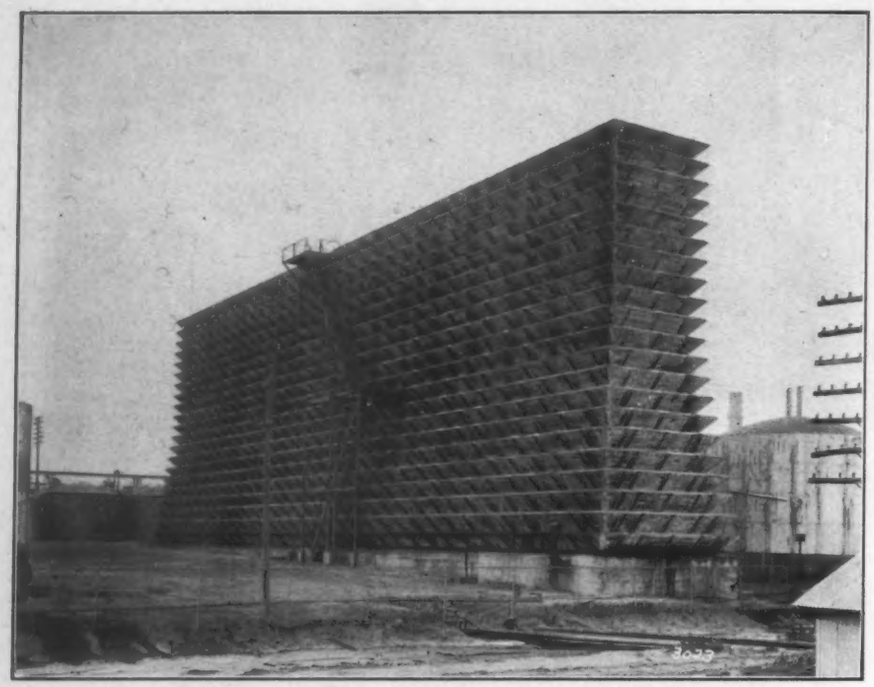
Filtration

Bleaching Action of Fuller's Earth on Oils—(Neumann and Kober. Z. Angew. Chem. 40 337 (1927). *Brit. Chem. Abs. -B, July* 8, 1927, *p.* 493.) The bleaching value of an earth was determined by a standardized method of agitating a given quantity of the earth with an oil, filtering, and examining the clarified oil colorimetrically. As color standards, 0.05 per cent. solutions of Fat-soluble Yellow AT, R, or 3R in paraffin oil are quite suitable. With vegetable oils, earths heated at 600 degrees give the greatest bleaching effect; but for mineral oils containing dyestuffs, untreated earths are more effective. This is explained by assuming that the unheated earths have the greater surface area and the smaller pores, capable of retaining the crystalloid particles of the dyestuffs; while the pores enlarged by heating and driving out of the water can take in the colloidal matter, e.g., mucilage and albumin, of vegetable oils. The bleaching of an oil is attained by using 2-4 per cent. by weight of earth. Attempting to correlate bleaching activity and chemical constitution, the authors determined water loss and soluble alumina and iron oxide in earths heated for definite periods at temperatures up to 1000 degrees. With five proprietary earths the water loss rose sharply to about 12 per cent. between 500 degrees and 600 degrees, and the soluble oxides reached a maximum between the same temperatures, but decreased rapidly between 800 degrees and 1000. In two cases the water loss rose steadily from 100 degrees to 900 degrees, and in one of these, an American earth, the soluble oxides were fairly constant at 8 per cent. as high as 800 degrees. These earths, with the exception of the American, gave the maximum bleaching effect on soya bean oil after treatment at 600 degrees, this property increasing rapidly after heating at 100-300 degrees and falling again at 800 degrees. The American oil gave its maximum effect at 300 degrees.

It is concluded that all the observed phenomena can be adequately explained on a physical-absorption basis.

Treatment

New Ways of Purifying Oil. Stegemann. (*Z. Angew Chem., June* 9, 1928, *p.* 626. *Libr. Bull., U. O. P. Co., Vol.* 3, *No.* 27.) Petroleum, brown coal tar oil, and crude benzol can be refined successfully with hydrofluoric acid. More or less hydrofluoric acid is required, depending on the purpose to which the oil is applied. Very favorable results are obtained by treating the oil at ordinary temperature with 3-4 per cent. of the acid for four hours. A crude benzol having a bromine number of 6 and sulfur content of 0.36 per cent. after this treatment scarcely should any reaction with sulpuric acid. the bromine number dropped to 0.2, organic sulfur was found only in traces, and Sulfur A could not be detected. Even with 1 per cent. of hydrofluoric acid a



One of our Special Atmospheric Cooling Towers installed at an Oklahoma refinery. This is the largest *single* Atmospheric Cooling Tower in the world, as far as we know.

Buy a Cooling Tower On Which You Can Depend

THE lowest priced cooling tower you can buy is apt to prove the most expensive in the long run. Buy a cooling tower on which you can depend and you have successfully solved your water cooling problems.

In many cases a correctly designed cooling tower or spray pond will return its entire cost during the first year or so. With responsible firms we are ready and willing to enter into an agreement whereby the cost of the tower is spread over a year or more so that the payments can be made out of the savings effected. Our nearest representative will be very glad to explain this plan to you in detail.

THE COOLING TOWER COMPANY, Inc.
15 JOHN STREET NEW YORK

Houston, Texas
The J. A. Rossiter Co.,
410 Union Nat'l Bank Building

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Tulsa, Okla.,
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Announcement

We desire to bring to your attention the fact that we have added to our well established line of equipment a department specializing in Heat Exchangers, Heaters, Coolers, Reflux Condensers, Partial Condensers, and similar apparatus particularly adaptable to the Oil Refining and Allied Industries.

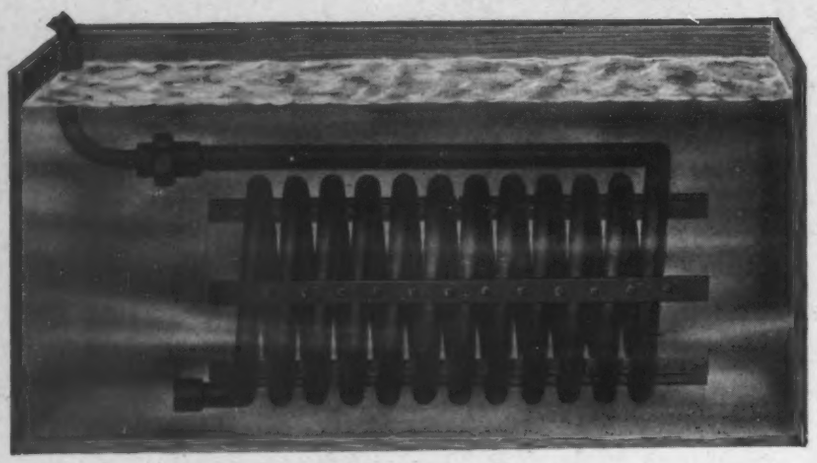
The thirty years experience of the J. P. Devine Manufacturing Company, Inc., in the manufacture of Condensers, Heaters, Evaporators, Dryers, Fractionating and Vacuum Apparatus, together with their extensive shop facilities and engineering force, places them in a position second to none. The well earned reputation of the Devine Company has been built up, not only by the design and excellence of its output, but also through the service it renders its customers. Our shop and equipment are the very latest and places us in a position to not only supply high-class equipment, but make prompt deliveries.

The Devine Company specializes in heating, cooling, condensing, evaporating, drying and fractionating equipment. We would like to have the opportunity of securing your business along these lines, and ask that you place us on your list so that whenever you are in the market for this class of equipment we will receive your specifications and have the opportunity of quoting upon your requirements.

J. P. Devine Manufacturing Co., Inc.

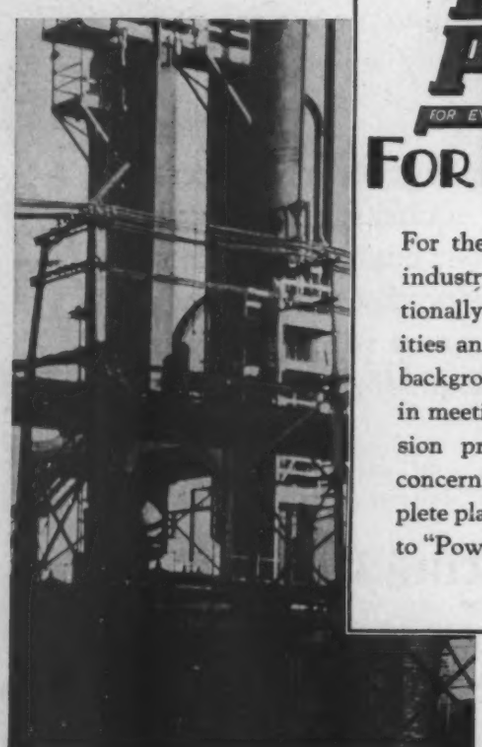
Buffalo, New York

PITTSBURGH
a air p pressure t tested COILS



Tested! Into a deep tank of water, the air hose coupled on, pressure opened up—and if there is a leak in a mile of Pittsburgh Coil it is caught quick, it never reaches your plant. Blue print or rough sketch brings quotation.

Pittsburgh Pipe Coil & Bending Co.
P. O. Box 975, Pittsburgh, Pa.



Power Piping
FOR EVERY OIL FIELD NEED

FOR REFINERIES

For the complex needs of the industry this company is exceptionally equipped both in facilities and in personnel, with a background of vast experience in meeting the power transmission problems of leading oil concerns. Whether it's a complete plant or a fitting, put it up to "Power Piping."

O 1

POWER PIPING CO. PITTSBURGH

benzol can be obtained which is entirely stable to light and air. The hydrofluoric acid boils at 20 degrees C. and is easily separated from the treated oil by warming. The residue obtained is a very valuable pitch instead of the acid empyreumatic resins formed in sulfuric-acid treatment; the residues from the sulfuric-acid treatment of benzol are of no value and those from pertoleum oils require to be neutralized before they can be used. Refining losses are much smaller with hydrofluoric than with sulfuric-acid treatment.

Another purifying process especially suitable for benzol depends on the dehydrogenating action of sulfur. A crude benzol with the bromine number 6 heated to 250 degrees C. for two hours with 1 per cent. of sulfur then showed a bromine number of 0.6, and gave no reaction for Sulfur A. The refining loss is very small and the residue is again a valuable pitch.

The Chemical Processes in Sulfuric-Acid Refining. Tausz. (*Z. Angew Chem. June* 9, 1928, *p.* 628. *Libr. Bull., U. O. P. Co., Vol.* 3, *No.* 27.) The very general use of sulfuric acid for refining is responsible for considerable losses of valuable oils. Little is known of the chemical processes which take place in this method of refining. The author undertook to study the action of the sulfuric acid upon individual pure unsaturated hydrocarbons, but the course of reaction is not the same in mixtures as with single pure compounds. The fact is established that the sulfuric acid may serve to condense two differing compounds. Sulfur compounds have the property of yielding dye-like compounds by condensing with unsaturated hydrocarbons. Sulfuric acid acting upon thiophene and cyclopentadiene gives a red dye, upon thiophene and cyclohexene, a green. All the dyes, whatever their color in the free state, are soluble in sulfuric acid with orange-yellow color. Oxygen compounds also give colored compounds. These may be formed by the autoxidation of unsaturates.

In general, the color which appears when benzines and benzols are shaken with sulfuric acid is the result of a condensation of sulfur or oxygen compounds with olefinic hydrocarbons. With longer action of the air the dye goes over to the well-known acid resin.

Acid Sludge from Oil Fractions from Emba Crude Oil. Obryadchikov. (*Neftyanoye Khoz.*, 14, 360-2 (1928). *C. A. Vol.* 22, *No.* 12, *p.* 2264.) The acid sludge was washed with water, the residue heated in an oil bath to 130 degrees to 150 degrees C. At this temperature the coke separated from the tar. The separated tar was treated with air at 300 degrees C. and asphalts melting at 80 degrees, 60 degrees and 30 degrees C. were obtained. At 130 degrees C. there were formed water and gas 2.15 per cent., asphalt 56.6 per cent., coke 44.25 per cent.; at 150 degrees C. water and gas 6.03 per cent., asphalt 51.82 per cent., coke 42.15 per cent.; at 170 degrees C. water and gas 13.94 per cent., asphalt 50.87 per cent., coke 35.19 per cent. The coke mixed with some cheap oil could be used as fuel.

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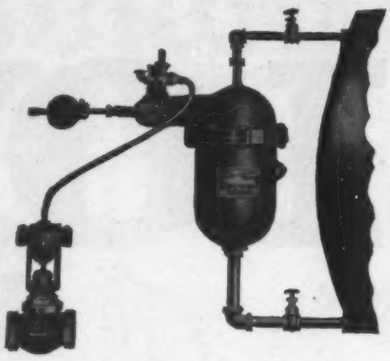

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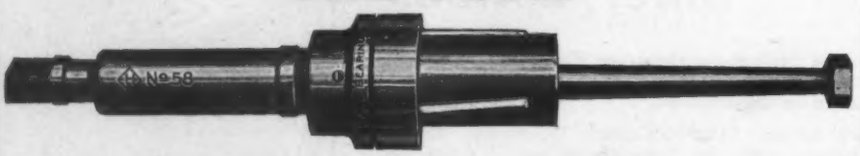

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Treatment of Bottoms from the Lubricating-Oil Fraction of Emba Crude Oil. Chernozhukov. (*Neftyanoye Khoz.* 13, 188 (1927). *C. A., Vol.* 22, *No.* 12, *p.* 2265.) The heavy bottoms were treated with a mixture of two parts of sulfuric acid and one part of naphthenic acids left from treatment of engine oils. Forty-five per cent. of oil could be separated this way.

Refrigeration

Increasing Efficiency of Refrigerating Systems. Hilger (*Ice and Refrig., Vol.* 74, *No.* 4, *April,* 1928, *pp.* 403-406.) Apparatus and methods for increasing efficiency of refrigerating machines; relationship of temperatures and pressures of working refrigerants; effect of non-condensable gases; control and regulation of evaporators; describes and illustrates various apparatus and methods which may be used to increase operating efficiency of compression refrigerating system.

Of Particular Interest to the Refinery and Chemical Engineer

Corrosion

The Practical Problems of Corrosion. Part III. The Formation of Rust and Its Consequences. Evans. (*J. S. C. I., March* 9, 1928, *p.* 1928, *p.* 55*T*, *Libr., Bull., U. O. P. Co., Vol.* 3, *No.* 13, *pp.* 125-26.) Rust formed under water is composed mainly of ferric hydroxide or hydrated ferric oxide, yellowish-brown, and a greenish ferrosoferric hydroxide, perhaps $Fe_3O_4.xH_2O$, underneath where oxygen has less ready access. The rust usually contains also small quantities of chloride, sulfate, phosphate (from the metal) and carbonate. The chlorides and sulfates are often described as basic, but there may be merely ferric hydroxide with absorbed ferric chloride and sulfate.

The great bulkiness of freshly formed rust or rust sludge is much more important than its chemical composition. This sludge even after a month's settling may occupy a volume from 130 to 320 times the volume of the iron destroyed; which explains the readiness with which pipes are choked up by rust. Considerable water is probably enmeshed with the ferric compounds, but even the dry rust occupies more space than the original iron. Sheet aluminum when placed in water containing chlorides and bicarbonates was corroded in pores just under the surface and blisters were raised by the voluminous aluminum hydroxide formed. Cast iron has been known to crack from the action of concentrated sulfuric acid; this was thought to be due to the formation of crystalline iron sulfate in pores, the growing crystals expanding with sufficient force to break the iron. Where iron has been buried in concrete and stray electric currents have led to corrosion of the iron, the concrete has in some cases been shattered. In one instance the iron stirrups originally ⅛-inch thick had become covered with rust 3/16-¼-inches thick, though the uncorroded iron was till 1/16-inch in thickness. In another the rust had not

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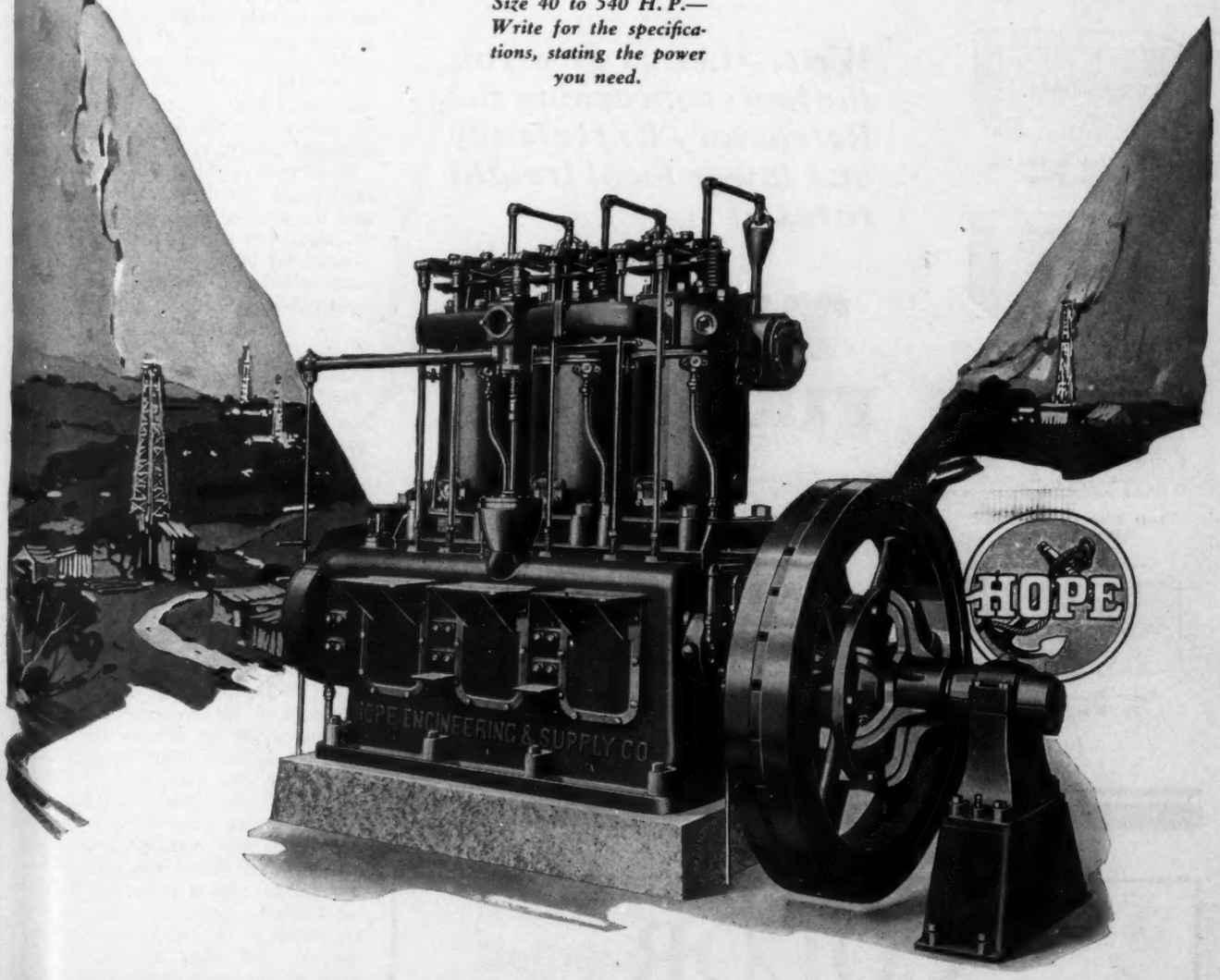

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been precipitated immediately on the iron but in the concrete itself.

Iron immersed in most waters will be progressively corroded at the anode if from any cause an electric current passes, and the first product of chemical action will be ferrous, soluble, and not deposited until it has passed into a region richer in oxygen where it becomes ferric and insoluble.

Calcium and magnesium sulfates in hard water even in very low concentrations shield immersed iron almost completely from oxygen and corrosion is much slower in such waters than in distilled water. Magnesium chloride is less corrosive than sodium chloride at low temperatures but at the boiling point the hydro chloric acid produced by the hydrolysis of the magnesium chloride is corrosive even in absence of oxygen.

The corrosion which results in the formation of voluminous precipitates in pores is most apt to occur in metals which are under tension; and measurements have shown that with alternating stresses the endurance limit is reduced by this kind of corrosion much more than the tensile strength, because cracks form and spread.

In atmospheric corrosion, therefore, the mechanical strength, the micro-structure, and the internal and external stresses on the metal are important factors. Yet the present-day laboratory corrosion tests take account only of chemical or at the most electrochemical action.

Oil Flow in Pipes

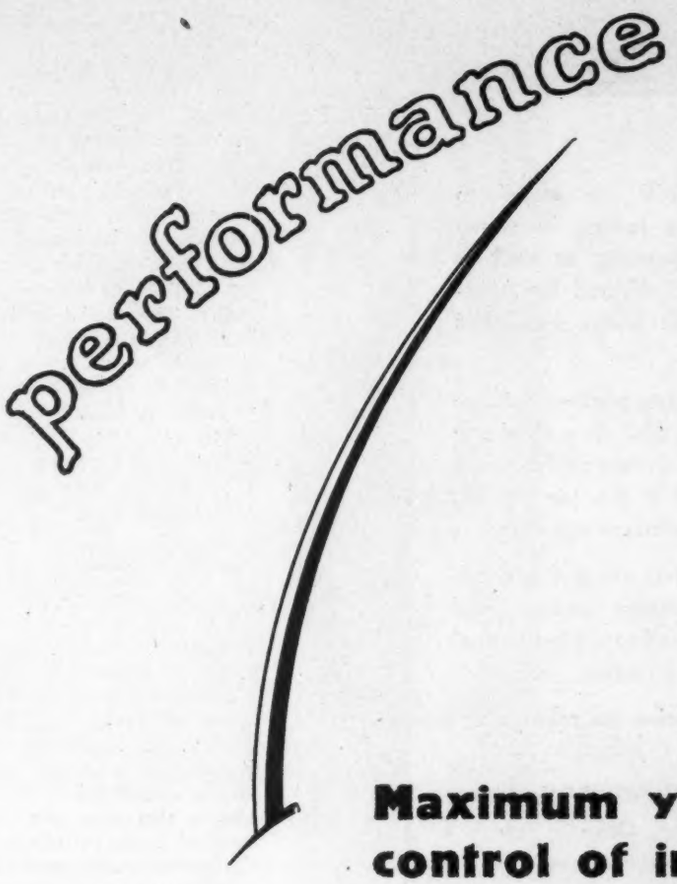
The Flow of Viscous Liquids Through Pipes. (*Engineering, Vol.* 125, *No.* 250, *April* 27, 1928, *pp.* 498-499.) Oils of various degrees of viscosity are frequently pumped through pipes to ships or to distant storage tanks; to determine power required in given case and to choose most economical pumping plants and pipes, frictional resistance encountered by liquids when so transported must be known; presents formulas and charts by C. H. Lee for determining viscous turbulent flow, and describes method of using them.

OF PARTICULAR INTEREST TO THE CHEMIST

Testing and Analysis

Improved Apparatus for Determining Melting Point by Electric Signal. Wick and Barchfield. (*Die Chem. Fab., May* 16, 1928, *p.* 281. *Libr. Bull, U. O. P. Co., Vol.* 3, *No.* 26.) The melting-point block is set in a ring attached to a vertical support. The tube containing the sample to be melted is placed in the block and on the sample rests a glass rod which holds up a contact arm, one end of which is connected to a battery attached on the support; the other is free, but for the glass, to drop upon the tip of a micrometer screw in the outer side of the ring support beyond the block. The melting of the sample allows the glass rod to drop, the contact arm then drops on the tip of the micrometer screw, the electric circuit is closed, and a signal is given.

The Determination of Small Quantities of Water in Mineral Oils. Boller. (*Petroleum, Feb.* 10, 1927, *Chim, et. ind., Feb.* 1928, *p.* 243, *Libr. Bull., U. O. P. Co., Vol.* 3, *No.* 13, *p.* 127.) A current of nitrogen or hydrogen is passed at the rate of 10-15 bubbles per 10 seconds through 100 g. of



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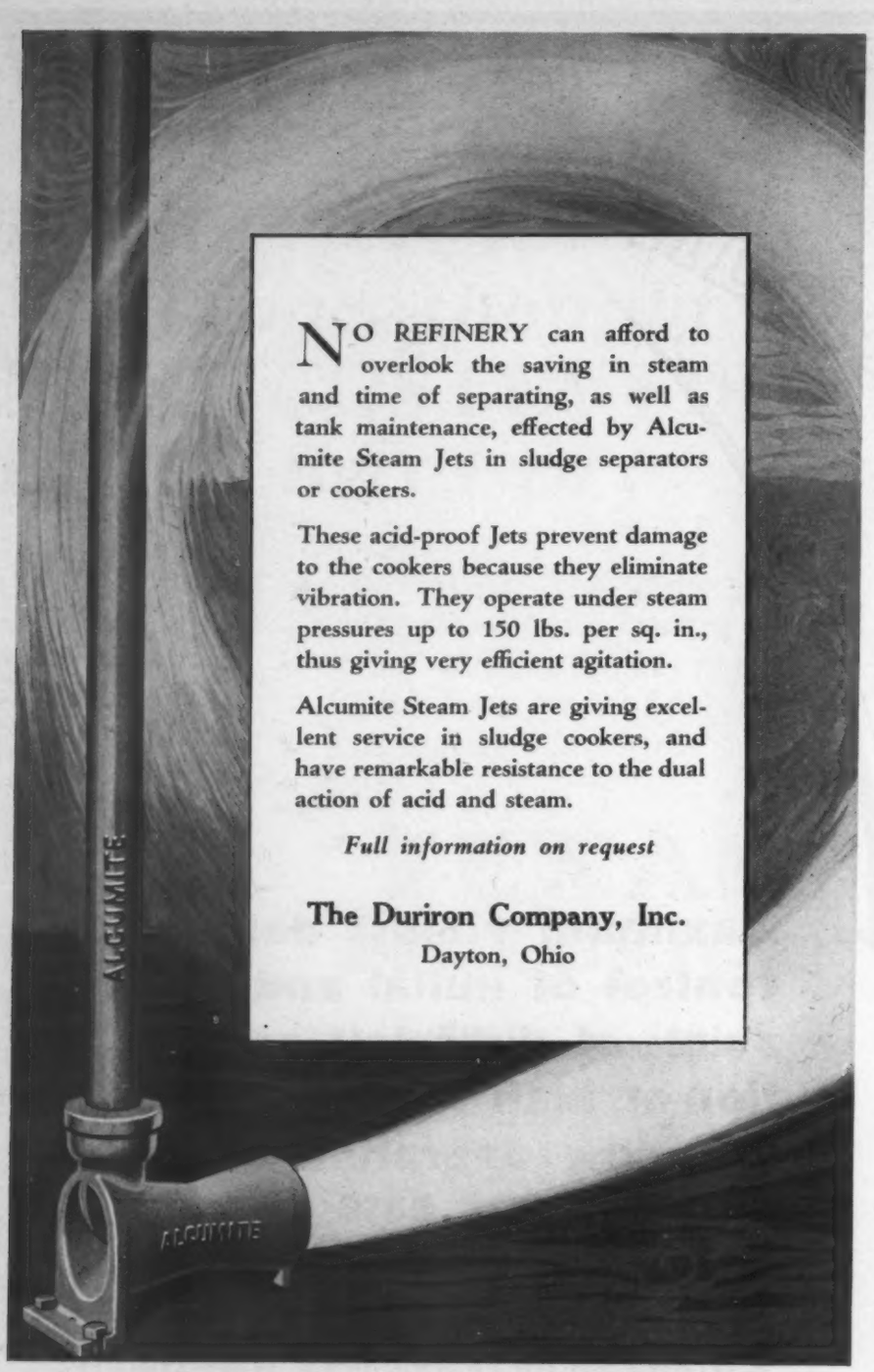

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mineral oil heated on an oil bath to 130-140 degrees, a tube containing calcium carbide in grains 3-4 mm. in diam., heated to 130-140 degrees, and an absorption tube containing cuprous ammoniacal solution. The acetylene produced by the action of the water upon the calcium carbide forms cuprous carbide as a precipitate in the cuprous solution which may be determined either (a) volumetrically or (b) gravimetrically, after filtering and washing with water containing 2 per cent. of ammonia.

(a) The precipitate is dissolved in a solution of ferric sulfate and the ferrous sulfate formed corresponding to the cuprous carbide is titrated with 0.1 N. permanganate of which 1 c.c. is equivalent to 1.802 mg. H_2O.

(b) The precipitate is dissolved in dilute sulfuric acid and the copper is precipitated with hydrogen sulfide and weighed as copper oxide, of which 0.1 g. = 0.2264 g. H_2O.

The method is applicable to very small quantities of water for which Marcusson's xylol method does not give sufficiently accurate readings.

Results of Research and Investigational Work

The Separation of the Components of Petroleum. (Part V. Edge Filtration. Part I. The Isolation of Waxes from Untopped Crude Oil without Pyrolysis. Gordon and Marshall. Jour. Soc. Chem. Ind. July 15, 1927, *p.* 304T.) As the title indicates, this is the fifth of a series of reports upon the general subject. It was previously shown that wax probably exists in the original crude petroleum instead of being formed by some pyrolytic process in distillation; and that it is kept from separating out on chilling by the protective colloidal action of the heavy petroleum hydrocarbons present. The aim of this new work was to show that the wax substances can be made to separate out by the use merely of edge filtration and of solvents.

An untopped Persian crude was used for the experiments, with a laboratory model of the Hele-Shaw stream-line filter. Four filtrations were made of one sample, and from the second residue dissolved in carbon tetrachloride a wax was obtained. A later series of filtrations showed that not only was paraffin material separable from the asphaltic residue, but so, too, was wax also present in solution in the light components of the filtrate from which it could be separated by evaporation.

All the waxes separated appeared to be amorphous, though quite free from oil. They were hard and brittle and resembled plaster of Paris that has set.

Some preliminary tests upon the effect of topping with steam before filtering reduced the amount of residue obtainable by edge filtration. This raises the question, did the steam destroy some of the colloidal material, are the light fractions necessary to help separate the colloids, or is the difference merely the result of change in viscosity or surface tension? In answer, an investigation is being made of the effect on the crude oil of removing

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the light fractions by a method which cannot involve pyrolysis.

The Separation of the Components of Petroleum. Part VI. The Action of Glacial Acetic Acid. (*Gordon and Kerry. J. S. C. I. Nov.* 18, 1927 *p.* 430*T. Libr. Bull., U. O. P. Co., Vol.* 2, *No.* 48, *pp.* 433-34., *No.* 49, *pp.* 443-44). Untopped Persian petroleum with a sp. gr. of 0.838, and an asphalt content of about 3 per cent (Kissling's method) was employed for these studies. Glacial acetic acid extraction gave a red solution containing very volatile components which crawled over the edge of the dish and in a flask, even, escaped through the cork. This did not occur with absolute alcohol as the solvent. The black, viscous residue when filtered gave (1) a black, waxy paste and (2) an "asphaltic residue." Better results, easier working, were yielded by strictly anhydrous acid.

On the second trial 300 cc. of insoluble oil was left from 600 cc. of crude; this contained 30.4 g. of asphaltic residue. The acid-soluble portion was fractioned by adding successive portions of water. Heavy loss of volatiles took place in dehydrating. All fractions flashed at about 50°.

The acid-insoluble portion had the same flash point. Its color was not removed by bone char or fuller's earth. It was separated into two fractions by hot acetone; when cold, acetone gave no separation. Asphaltic residue, dark-brown, waxy paste, melting point 35°; soluble in carbon bisulfide, carbon tetrachloride, benzene, pinene; partially soluble in ether, chloroform, hot glacial acetic acid, and hot alcohol; not in cold alcohol.

Yields of asphaltic residue from crude by the use of other solvents than acetic acid: amyl alcohol, 3.2 g. from 100 c. c.; absolute alcohol, 3 g.; by absolute alcohol added to ether solution, 4 g.; chloroform, 1.5 g.; ether, 4.3 g.; by filtration after topping with open steam, 2.4 g. These were all similar in appearance, gravity, and solubilities.

It is probable that the asphaltic material is in suspension in the petroleum and that the effect of the solvents is to remove the protective membrane of oil, allowing some of the asphaltic, waxy material to drop out of suspension. The wax as filtered off may be amorphous or may have crystallized. The effect of steam topping is also to coagulate a portion of the asphaltic substance—not necessarily a pyrolytic action. In some previous work, less asphaltic residue was obtained from steam-topped oil than from the original crude, by edge filtration. The pack had to be loosened in order for the topped oil to filter and apparently the colloid particles passed between the sheets of paper and were not recovered. Had steam coagulation thrown out more "asphalt" than was taken out of the crude by edge filtration, the balance, of course, would have been on the other side.

1000 c. c. of oil + 2500 c. c. glacial acetic acid gave a solution which was fractionated by successive additions of water into five, increasingly-heavier fractions

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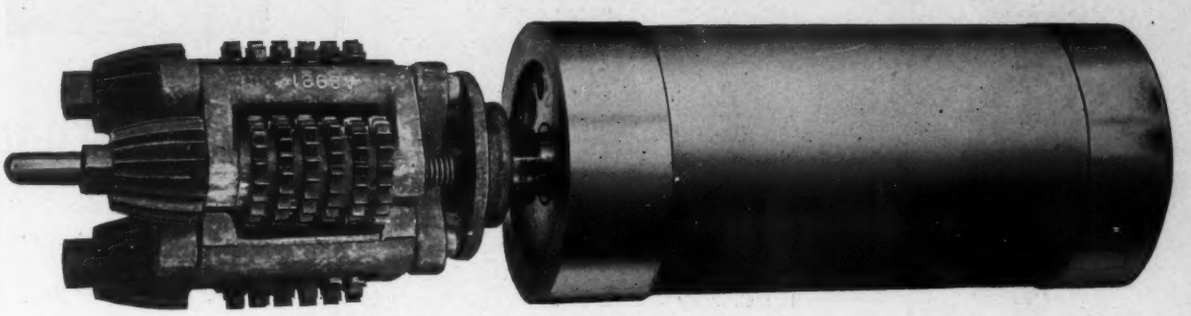

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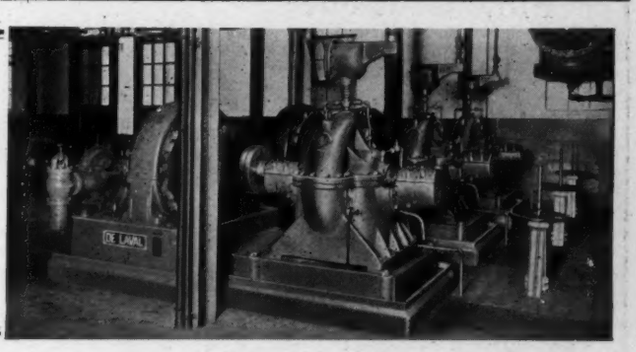

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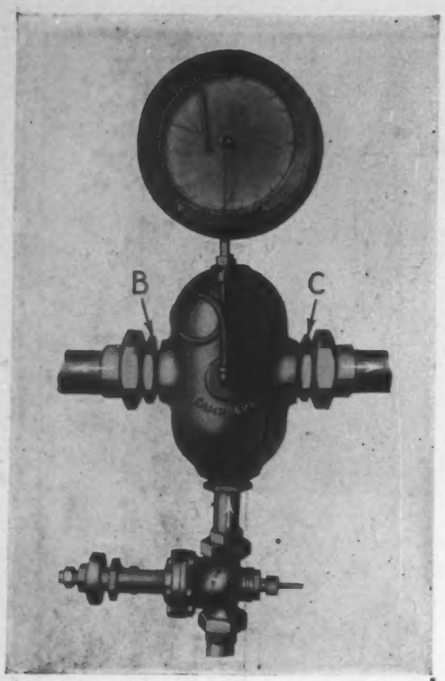

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Acid-insoluble oil dissolved in ether, fractionated by successive additions of absolute alcohol. Heaviest fractions came out first. After adding excess of alcohol, water was added; this threw out an oil of high gravity, marked fluorescence, and very soluble in alcohol. The preceding six fractions were progressively lighter in color and more fluorescent.

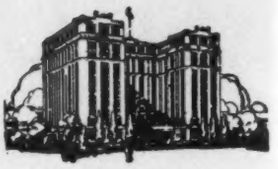
Asphalt insoluble in acetic acid dissolved in carbon bisulphide, and was decolorized by bone char; the refined asphalt cooled in carbon bisulphide was made to yield several crops of wax crystals. A solution of the asphalt in carbon tetrachloride was passed through bone char and a brown oil was extracted from the char by carbon bisulfide. Ether and chloroform each dissolved about four-fifths of the refined asphalt. The solution in each case contained white wax and brown oil, which was probably present as a fine and stable emulsion.

100 c. c. of crude oil was also fractionated from ether solution by adding, first, alcohol and then water. The first fractions were black or brown-black, but became progressively lighter and possessed fluorescence.

* * *

New Uses

New Synthetic Oils. Wulff. (*Z. angew Chem., June* 9, 1928, *p.* 626. *Libr. Bull., U. O. P. Co., Vol.* 3, *No.* 27). A process is described for making high-grade lubricants from aromatic hydrocarbons and olefines with the aid of catalyzers of the Friedel-Craft reaction. By the action of olefines, as ethylene, propylene, butylene, and higher olefines upon benzol, toluol, naphthalene, and oils which contain aromatics, for instance coal-tar oil, and tetralin, in presence of halides of aluminum, iron, or boron, at temperatures up to 350° C., oils are obtained which resemble the American lubricating oils. For example, a rotating bomb of 4-5 liters capacity is charged with 700 g. of naphthalene, 40 g. aluminum chloride and ethylene to a pressure of 80-85 atmos., and heated and rotated. Pressure rises to about 120 atmos. and then drops again, as heating is continued to 240° C. When the contents of the bomb cool to room temperature the pressure is 10-15 atmos. Yield, 1300 g. of dark brown fluorescent oil. By steam-distilling, 1-2 per cent of lime being added to bind the hydrochloric acid, 10-15 per cent of ethyl and diethyl naphthlene, principally, goes over. The lowest fractions are ethyl and butyl chloride, with small quantities of benzine. The crude oil treated with steam has a viscosity of 4.8°E. at 50° C. (122° F.), flash point 150° C., tar number 0.1-0.2 per cent. By distilling, light oils for other purposes can be obtained, for instance, a bright light-green transformer oil, flash point 150°, viscosity 6°E. at 20°, cold point below -15°, tar number 0.09-0.10 per cent, sp. gr. 0.95. This distillate can be ob-



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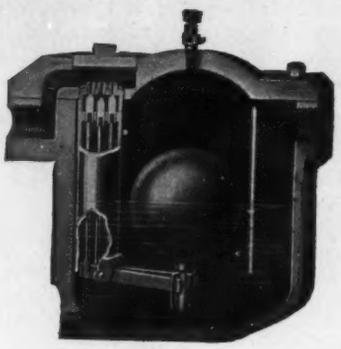

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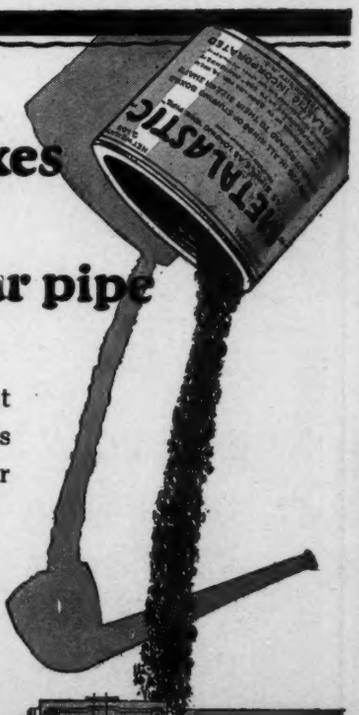

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tained in quantities of 70-75 per cent. By distilling off the low-boiling constituents up to 180° at 7 mm., 70 per cent of a highly viscous lubricant is obtained 8.6°E. at 50°, 3.5 at 80°. On refining with concentrated sulfuric acid the crude oil loses 2-3 per cent, and the tar number drops to 0.02-0.04 per cent The reaction products may be determined by the reaction temperature or the quantity of ethylene. When boron fluoride is the catalyzer, the oils formed contain more hydrogen than with aluminum chloride. Otherwise, the oils have similar technical properties. The addition of small quantities of halogen acids and of certain metal chlorides accelerates the Friedel-Craft reaction. The halide of boron can be recovered very easily. The quantity of catalyzer to be added is 3-4 per cent.

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Natural Gasoline Industry

Recoverable Hydrocarbons from Natural Gas and their Relation to the Composition of Commercial Motor Fuel. (Coleman. Meeting of Amer. Inst. of Chem. Engrs., St. Louis, Dec. 5-8, 1927. Libr. Bull., U. O. P. Co., Vol. 2, No. 50, pp. 451-52). The first casinghead gas brought in is lean; that is, it consists mainly of methane, ethane, and propane. As the pressure in the gas reservoir gradually drops, larger quantities of butane, pentane, and then the higher hydrocarbons are found in the mixture. The question of how much butane should be held in the natural-gas gasoline depends upon (1) its value locally as a fuel, (2) the cost of recovering it, and (3) the relation, under existing conditions of storage and distribution, between butane content and evaporation loss.

In the absence of definite information upon the third point the Humble Oil and Refining Company determined vapor pressures and evaporation losses in gasoline containing known amounts of butane, as the gasoline was stored at the Baytown (Texas) refinery and transferred from storage to the city of Houston and to the local filling stations. When per cent losses were plotted against vapor pressures, the curve was nearly a straight line in the range of vapor pressure of practically all present-day motor gasolines, namely, from 200 to 400 mm.; above 400 mm. the curve tends to become asymptotic and probably does become so at 760 mm.

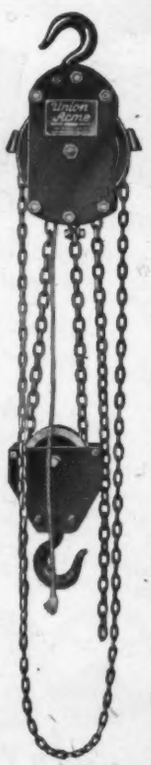
This makes it possible to calculate percentage losses at other vapor pressures, *under similar conditions* of storage and transfer. When the data were plotted on semi-log paper, the curve was a straight line. The percentages of propane present were from Raoult's law and a knowledge of the percentages of propane and butane in the mixtures calculated over to the percentages of butane which would give the same vapor pressures. These equivalents were added to the actual butane pressures. It was found that the losses due to raising the butane content about 8 per cent amounted to only about 1.6 per cent over those taking place without added butane; a fact which under no more severe evaportation conditions economically favors the addition of the volatile constituents so desirable from the standpoint of the consumer.

Another set of conditions would require the determination of but one point on the vapor pressure-evaporation loss curve.

The Engler distillation in no way reflects the absolute vapor pressure of gasoline. The I. B. P. of 60° F. is 26° above the boiling point of butane, which in some cases contributes 50 per cent of the total vapor pressure. Whereas under recovery by simple condensation the quantity over at 212° F. gives a rough measure of the pentane, hexane, etc., and by inference butane, with rectification no such clue is afforded by the Engler distillation. The gasoline industry should establish a specification for vapor pressure.

Announcement is made of the removal of the offices of Merco Nordstrom Valve Company, the Merrill Company and the Alloys Company in San Francisco, from their Second Street address to the top floor of the building at 343 Sansome Street. Offices occupying approximately 6000 square feet have been leased for a long period of years and a complete laboratory has been installed on the roof of the building to carry out experimental work. The Merrill Company occupied its old location for 15 years and the Valve Company was established as a subsidiary corporation in 1924. Prior to that time it was operated as a department of the Merrill Company. Announcement is also made that the Chicago office of the Valve Company will be removed in October from the People's Gas Building to the Midland Building.

The Superheater Company, 17 East 42nd Street, New York City, is distributing a publication called "Steam Tables," which give the properties of saturated and superheated steam from 0.0886 to 3300 pounds, absolute pressure. The tables have been compiled from Marks and Davis, A. S. M. E. Committee on Properties of Steam and Extension of the Steam Tables, and Stodola, and are said to be in line with the present-day demand for data on the higher pressures. A convenient heat- entrophy diagram is included. Copies may be obtained by request from the company.



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